A STUDY OF
SHORT TEST AND CHARGE RETENTION TEST METHODS FOR NICKEL-CADMIUM SPACECRAFT CELLS

FINAL REPORT

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TRW SYSTEMS GROUP

ONE SPACE PARK, REDONDO BEACH, CALIFORNIA 90278

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ABSTRACT

Three different methods for testing nickel-cadmium cells for internal shorts and charge retention were studied. Included were (a) open circuit voltage decay after a brief charge, (b) open circuit voltage recovery after shorting, and (c) open circuit voltage decay and capacity loss after a full charge, with emphasis on the first of these methods. The investigation included consideration of the effects of prior history, of conditioning cells prior to testing, and of various test method variables on the results of the tests. Sensitivity of the tests was calibrated in terms of equivalent external resistance. The results from the different methods were correlated. It was shown that a large number of variables may affect the results of these tests significantly. Recommendations are given for improved procedures to minimize these effects. It is concluded that the voltage decay after a brief charge and the voltage recovery methods are more sensitive than the charged stand method, and can detect an internal short equivalent to a resistance of about $(10^4/C)$ ohms where "C" is the numerical value of the capacity of the cell in ampere hours.

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

ILLUSTRATIONS

ILLUSTRATIONS (Continued)

ILLUSTRATIONS (Continued)

TABLES

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1. INTRODUCTION

This is the final technical report of a study of methods for testing nickel-cadmium cells for the presence of internal shorts or charge retention. The work was performed under JPL Contract No. 953649 as a supplement to a previous study of degradation of plates for nickel-cadmium cells.[1] The objectives of the present study were to clarify some of the factors controlling the open circuit voltage behavior during presently used tests, to determine the inherent sensitivity of the different methods, and to recommend improvements in test methods as necessary. The intent of the work was not to make an exhaustive theoretical study but to focus attention on those practical factors and procedural details that can most improve the reliability of these tests.

2. BACKGROUND

2.1 Origin of This Study

The study reported herein was undertaken after a series of erratic test results were obtained, early in 1974, which raised doubts about the validity and reliability of methods for detection of internal shorts in use at that time. Details of the test results referred to, and results from some additional testing designed to try to determine the underlying cause of the observed open-circuit voltage behavior, were presented in Reference 1. The original work reported in Reference 1 was concerned primarily with the application of prescribed test methods to program hardware, and with the question of flight-worthiness of the cells in question. Thus no investigation of the test methods, per se, was undertaken earlier. The study now reported on is an outgrowth of the above work.

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(1) "A study of Degradation of Plates for Nickel-Cadmium Spacecraft Cells," Final Report, W. R. Scott, JPL Contract No. 953649, June 1974.

2.2 Description of the Tests Investigated

Three types of tests were included in the study. These are referred to herein as (1) the "Voltage Decay Test"; (2) the "Voltage Recovery Test"; and (3) the "Charged Open Circuit Stand Test." The exact details of these tests differ among users. Typical procedures and a brief statement of the technical basis for each test are given below.

2.2.1 The "Voltage Decay" Test Procedure

The procedure for the Voltage Decay Test is usually as follows: After being shorted externally for some period of time, the cell is charged at a C/10 rate for 5 or 6 minutes, then allowed to stand on open circuit for 24 hours. At the end of that time, the terminal voltage is measured with a high-impedance voltmeter. The criterion of passing this test is a final voltage in the range from 1.15 to 1.20 volts.

The initial shorted stand period (which usually follows a discharge) prior to injecting the brief charge is intended to discharge the cell completely, or at least reduce the residual charge to negligible proportions. The charge added, which amounts to approximately 1 percent of the rated capacity of the cell, raises the open-circuit voltage to over 1.25 V (in a good cell). Then, over a 24 hour period, this open circuit voltage will decrease to a value below the acceptable level if there is an internal short. It is usually assumed that the decrease in voltage is due to electrochemical discharge of the cell with the shorting path acting as a load.

2.2.2 The "Voltage Recovery" Test Procedure

In the Voltage Recovery Test, the cell is charged fully, discharged to 1 volt at the C/2 rate, then further discharged to near zero volts with a resistor, and may or may not be shorted after that. The external load is then removed and the cell left on open circuit for up to 24 hours. The terminal voltage is then measured with a high-impedance voltmeter. The criterion of passing this test is an end voltage in the range from 1.17 to 1.20 volts.

This test procedure is based on the fact that the electrodes do not in fact become completely discharged (to the point where the open circuit

cell voltage remains near zero) after the cell has stood for, say, 16 hours with a low-resistance load following a charge and a discharge to 1 volt. Hence, when the external load is removed, the cell voltage tends to rise to approach the normal open circuit potential. As aptly described by Mauer (Reference 2), the cell under these conditions behaves like a large capacitor, being charged by the essentially constant limiting diffusion current from the residual charge on the electrodes. If the cell has an internal shorting path, the shorting resistance is in parallel with the capacitor and hence lowers the maximum voltage developed across it. In addition, the internal short will continue to discharge the electrodes during the "open circuit" stand period, thus eventually reducing the diffusion current and with it the maximum "open circuit" voltage produced.

2.2.3 The Charged Open Circuit Stand Test Procedure

In the Charged Stand Test, the capacity of the cell is first determined by a controlled charge and discharge cycle. The cell is then recharged fully and allowed to stand on open circuit for a number of days, usually about one week. At the end of that time, the open circuit voltage is measured, the cell is discharged, and capacity again determined. The difference between the capacities before and after the open circuit stand is taken as a measure of the ability of the cell to retain charge. A correction may or may not be made for capacity loss due to "self-discharge." The final open circuit voltages are compared and acceptable limits applied to the deviation from the average.

This test is based on the fact that in a positive-limited cell (as most sealed spacecraft cells are designed to be), the discharge capacity of the cell is a measure of the state of charge of the positive electrode. Hence any abnormal loss of charge of the positive electrode during the stand period should be reflected in a lower discharge capacity and a lower cell voltage at the end of the stand.

2.3 Purpose of Performing the Tests

Although the first two of the above three tests are referred to as "charge retention tests," they are in fact used primarily to detect the presence of internal shorting paths. The cell manufacturer may elect to perform such a test as a check on his manufacturing methods, or he

may be required by a customer to perform such a test as part of Quality Assurance testing. At this point in the life of a cell, it is assumed that any shorted behavior is due only to a manufacturing defect. Later on in the life of a cell such tests may be applied to determine whether any shorting paths have been produced by the effects of time and usage. The criteria of acceptability may be quite different for each of these cases, however.

As these tests have normally been performed (as described above) and with the treatment given to the data, they cannot distinguish between loss of charge due to chemical reactions within the cell and loss due to electrochemical discharge through an internal resistive path, as either phenomenon will cause the open-circuit voltage to be lower than normal after a time. One may argue that, in practice, it does not matter what the cause may be, and that cells showing such behavior should be rejected. If possible, however, it is valuable to be able to determine which cause is operative in order to be able to judge whether an open circuit voltage anomoly represents a random defect or whether it may be symptomatic of a defective lot of plates and/or cells.

3. ANALYSIS OF SHORT TEST METHODS

In this section the three methods used for short testing (or charge retention testing) as described in Section 2, are analyzed. The potential effects of prior history, of conditioning, and of variations in test parameters on test results are discussed in terms of the expected reproduceability and reliability of test results and sensitivity to internal shorting resistance. Reference is made to published and unpublished data available prior to the start of this study. As previously noted, primary emphasis is placed on evaluation of the Voltage Decay Test.

3.1 Potential Effects of Prior History and Conditioning

Prior history of cells may affect the results obtained on short testing in two ways; prior usage may cause a true internal short to form that was not present when the cell was new; and prior history may produce conditions within a cell that cause the test to give an erroneous

result, i.e., a short that is actually present may not be detected or the test may indicate a short when none is present. This study was concerned only with the latter type of prior history effects that may mask the true condition of the cell.

In general, the terminal voltage of a cell (as measured by a device which itself applies a negligible load to the cell) during periods when no load is attached externally, is a function of the positive and negative electrode potentials. The positive and negative electrode potentials under no-external-load conditions are in turn functions of their respective states of charge and prior histories. Finally, the states of charge as a function of stand time are functions of (a) the initial states of charge (at the time the cell external circuit is opened), (b) any internal electronically conductive path ("short") between the electrodes, and (c) the rates of chemical decomposition of charged material in the electrodes.

The initial state of charge of the nickel-oxide and cadmium electrodes depend on the cell design, on prior history, and on the nature of treatment of the cells immediately prior to performing open circuit tests. Cell design aspects of concern include total excess negative capacity installed, and the level of precharge (charged negative remaining after positive capacity is discharged) installed by the manufacturer. The possible impact of cell design is mentioned here only as a matter of interest, however, as the scope of the present study did not include evaluation of this variable. Those aspects of cell history, prior to testing, that probably affect test results include total age, number and type of cycles experienced, open circuit stand history and shorted stand history. Aspects of immediate pre-test treatment that could effect the results include whether or not the cell is conditioned, and the method of conditioning immediately prior to testing.

The general effects of life, cycling, open circuit stand, etc., on the electrochemical activity of the cadmium electrode are well known, and it is clear that almost any usage tends to decrease the activity of this electrode from its initial value. However, if sufficient excess negative capacity is installed, the effect of those variables on the open-circuit voltage should be minor.

The effect of those same factors on the activity of the positive electrode has received relatively little attention, presumably because the discharge performance of the positive over the usual operating range of the cell (above 1 volt at the terminals) is much less sensitive to use than is that of the negative. However, this may not be, and probably is not, the case after the positive has been discharged much further, as it is by placing a low resistance across the cell terminals for many hours following a discharge to 1 volt.

It is known, for example, that when a cell is new and has seen few cycles, the true state of charge of the positive is very low after a C/2 discharge to 1 volt. But after 50 or more cycles, much charged nickle active material remains after such a discharge. Thus, after many cycles, the positive electrode may take much longer to discharge to the same low state of charge than it did when the cell was new, and as a result the electrode potential behavior (after external shorting for a limited time) may be expected to be different in an older cell than in a newer cell. Thus it may be surmised that the behavior of older, more cycled cells may be expected to approach that of newer, less cycled cells after prolonged periods of standing with a low resistance or short connected across the terminals, as this treatment would reduce the positive electrode to the lowest possible state of charge. On the other hand, it is conceivable that the cell may be shorted for such a long time that it becomes discharged too far to give a meaningful short test. If so, this would indicate that some minimum amount of residual charged material must be present to stabilize the open-circuit electrode potentials. This possibility would impact the Voltage Recovery Test most strongly, particularly if the latter test is performed directly from a shorted condition without a prior charge-discharge cycle. Thus there is probably an optimum shorted stand period that should precede any short test, and each different test may have a different optimum time. Although determination of these optima was outside the scope of this study, some information was obtained by testing cells that had already been on shorted storage for various lengths of time.

In one sense, a shorted stand period prior to testing may be considered a form of "conditioning." Another form of conditioning is

charge-discharge cycling. Which of these will be the most useful form of conditioning may depend on the prior history involved i.e., standing with an external short (or low resistance load) may be best for cells that have seen much recent cycling, whereas one or more cycles may be the best conditioning for cells that have been started with external shorts for long periods. Furthermore, the use of both forms, in proper sequence, may produce even better results. Thus a heavily cycled cell may respond best to a shorted period followed by a cycle or two, whereas a cell after extended shorted storage may respond best to a few cycles followed by limited additional shorted stand. At best, however, such conditioning procedures should not be expected to erase all effects of prior history completely, but should minimize these effects and make the test results more uniform from cell to cell.

3.2 Effects of Test Procedure Variations

Another source of variation in short test results lies in the details of the test procedures themselves. Some users include some or all of those operations referred to above as "conditioning" as an integral part of the procedure, while others do not; the possible consequences of this type of procedure difference have been covered above. The potential effects of other procedural variations specific to the different short tests will now be considered.

3.2.1 Voltage Decay Test Procedure Variations

The type of test procedure variations that could affect the results of the Voltage Decay Test include:

a) Whether or not the cell is cycled immediately prior to the open circuit stand.

b) The number and type of cycles (if cycling is done).

c) The method of discharging to a low voltage, including the resistance used as a load and the time allowed on the resistor or the voltage to be reached;

d) Whether or not the cell is dead shorted (externally) and for how long, after the resistive discharge.

e) The charge rate used for injecting the test charge;

f) The ampere-hours (normalized to the cell capacity) passed during the test charge;

The potential effects of performing one or more cycles prior to performing the Voltage Decay Test were discussed above in the context of "conditioning." The main direct effect expected is a shift in the true state of charge of the positive electrode either upward or downward depending on how the cycling is done. This shift would tend to make the positive electrode potential more or less stable during the test and thus make the test less or more sensitive respectively, to shorting resistance than would be the case without prior cycling.

The one aspect of cycling that is considered by this writer to be most relevant to short testing is the ratio of charge to discharge ampere hours. At a given temperature and charge rate, a certain charge/discharge ratio will just maintain the true state of charge constant; higher ratios will increase the state of charge, while lower ratios will decrease it. It is believed that more satisfactory results will be obtained if the state of charge is always increased by such cycling, which, at room temperature, implies considerable overcharging. Such overcharging has the additional benefit of increasing the activity of the precharged negative capacity, thus producing a more stable negative electrode potential behavior.

Some information on the effect of different methods of charging during a single cycle preceding short testing was obtained from some work done by Gulton Industries (Reference 3) some years ago. In this work, which was directed to the Voltage Decay Test using an open circuit stand period of 24 hours, two different charge methods were tested (among other variables), namely, a C/2 charge for 2.5 hours, and a C/10 charge for 24 hours, both at room ambient temperature. The relevant data is shown plotted in Figures 1 and 2. For a 5 minute C/10 test charge, there was no significant difference between these two groups in the open circuit cell voltages after a 24 hour stand. There were obvious differences at lower test charge inputs, but these are not attributed to the prior charge-discharge cycle. Note that all pre-cycle discharges were done at a C/2 rate.

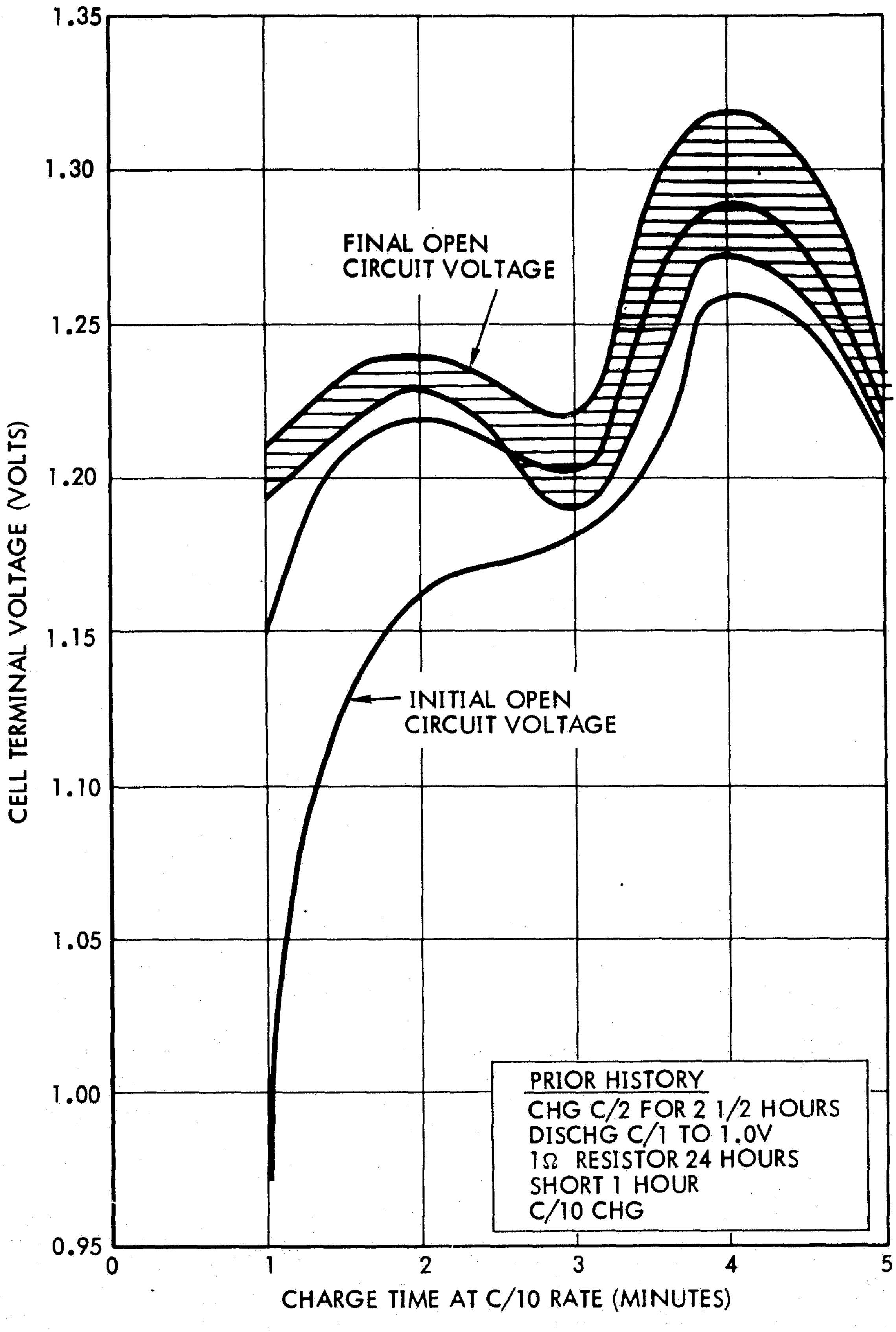


Figure 1. Voltage Decay Test Results, After a C/2 Conditioning Charge

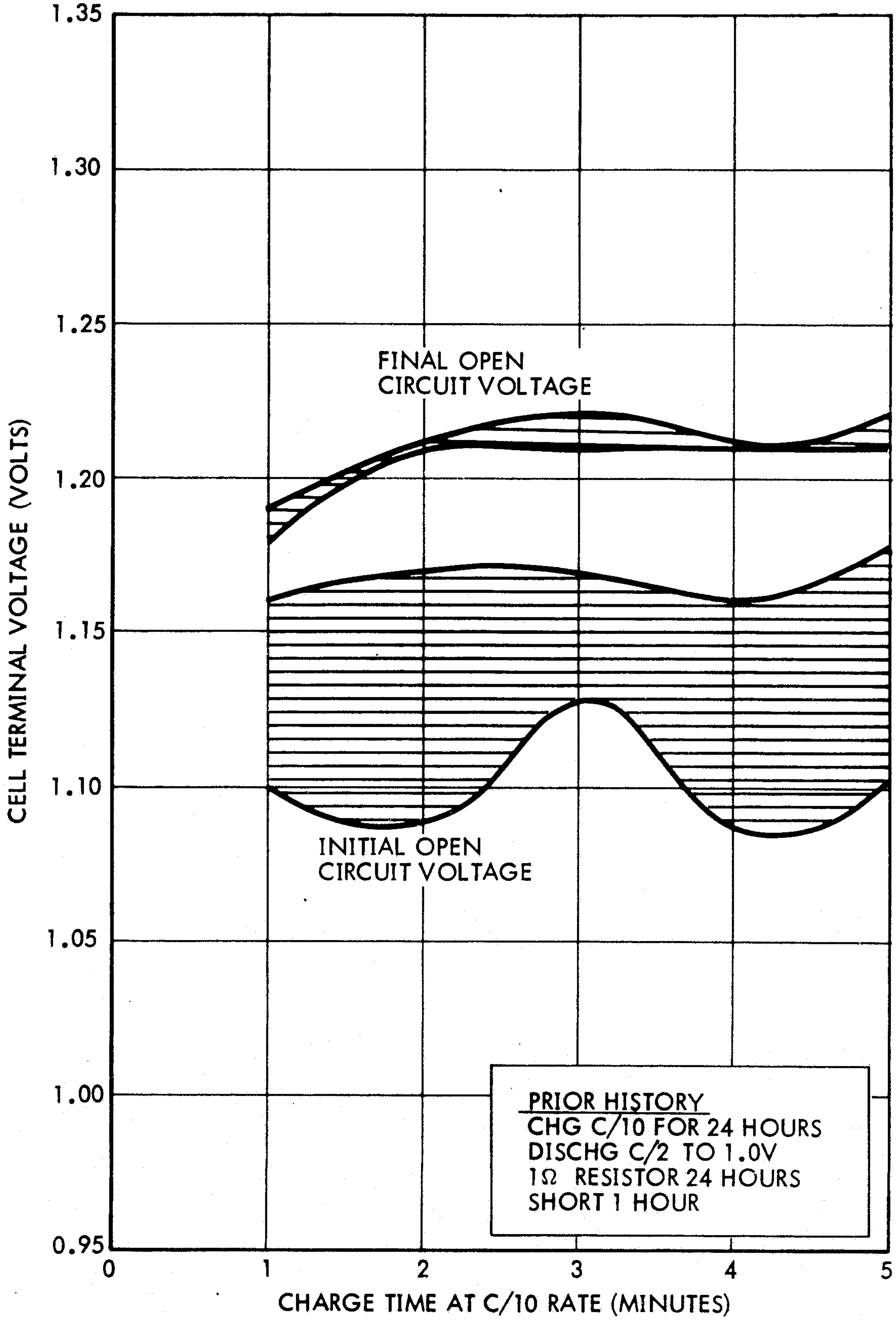


Figure 2. Voltage Decay Test Results, After a C/10 Conditioning Charge

In view of these results, it was decided to use the following set of conditions for testing of prior cycling: 25°C as the operating temperature, with a charge for 24 hours at the C/10 rate, followed by a discharge at the C/2 rate to 1.00 volt. This type of cycle is currently used by all cell manufacturers and most cell users for calibration purposes, and thus the use of these same conditions allows short testing to be fitted into overall test schedules with the least disturbance. It is this charge-discharge regime that was used through the experimental phase of this study. Other kinds of charge and discharge (above 1 volt) methods were not tested.

The effect of performing different numbers of cycles prior to open circuit testing is not known, and hence this variable was evaluated experimentally to a limited extent. Results on the Voltage Decay Test were compared after zero, one, and two cycles.

The size of the resistor used to allow the cell to discharge further after discharging at the C/2 rate to 1 volt, and the length of time on the resistor, will affect the true state of charge of the electrodes and hence may affect short test results. A related question is whether the use of dead shorts is necessary or beneficial after the cell has been on a low resistance load. The use of a one-ohm resistor for bringing cells down to a few tenths of a volt is common. However, the rate of discharge relative to the cell capacity decreases as the cell size (capacity) increases, using a one-ohm resistor (or any other single resistance value). The result is that, in a fixed time, large cells will be less discharged than small cells. Thus, in theory, the resistance for the load should be reduced in proportion to the cell capacity. In order to produce the same discharge characteristic as was obtained on the early 6 ah cells where the 1 ohm resistors were first used, the resistance should be kept close to 6/C ohms. The effect of departure from this formula was tested to a very limited extent during this study.

The length of time to allow on the resistor is also open to question. Most users now allow periods ranging from 8 to 24 hours, with 16 hours being most common. The rationale seems to be that (a) this time is convenient for an overnight stand, and (b) the terminal voltage is charging only very slowly after 16 hours, and hence a quasi-stable state has

been reached. There appears to be little knowledge of the number of ampere hours discharged during the time allowed, or of the current flowing at the end. Therefore some data on both of these points was obtained in the course of the testing during this study.

The relative merits of using a dead short after a resistive discharge was also considered. It has been stated (Maver, Reference 2) that the current flowing through a (low) resistance across a cell is independent of the resistance, below a cell voltage of about 0.6 volt. If true, this would imply that changing to dead shorts after a certain time on a low resistance is of no value. This possibility was investigated to a limited extent in the tests performed in this study.

Some data on the effect of variation of the total time allowed on a low resistance plus a short, in the range from 25 to 72 hours, was available (Reference 3), and is shown in Figures 3 through 6. Within the range of test charge input of from 3 to 5 minutes at a C/10 rate, no significant difference in voltage after 24 hours on open circuit is seen between the different short-down times indicated, with all voltages being close to 1.20 volts. It is interesting to note that after a 24 hour resistive discharge the open circuit voltage increased during the 24 hour stand, while after a 72 hour discharge the voltage decreased during the open circuit stand. Some light was shed on the reason for this difference during the present study.

The next procedure variable to be addressed is the charge rate used during the brief test charge injected prior to the open circuit stand. Most Voltage Decay testing has been done using the C/10 rate for this charge. From time to time when the results of this test have been erratic, the question has been raised as to whether the problem is due to a low charge efficiency at the C/10 rate. Prior to the present study, the only thing known that had been done to investigate this question was to make some comparative tests using a C/2 charge rate, shortening the charge time to give the same number of ampere hours. In general, these tests showed no significant difference between use of the two charge rates.

More recently, some data on charge efficiency at the two charge rates has been published by Bogner and Uchiyama (Reference 1). These

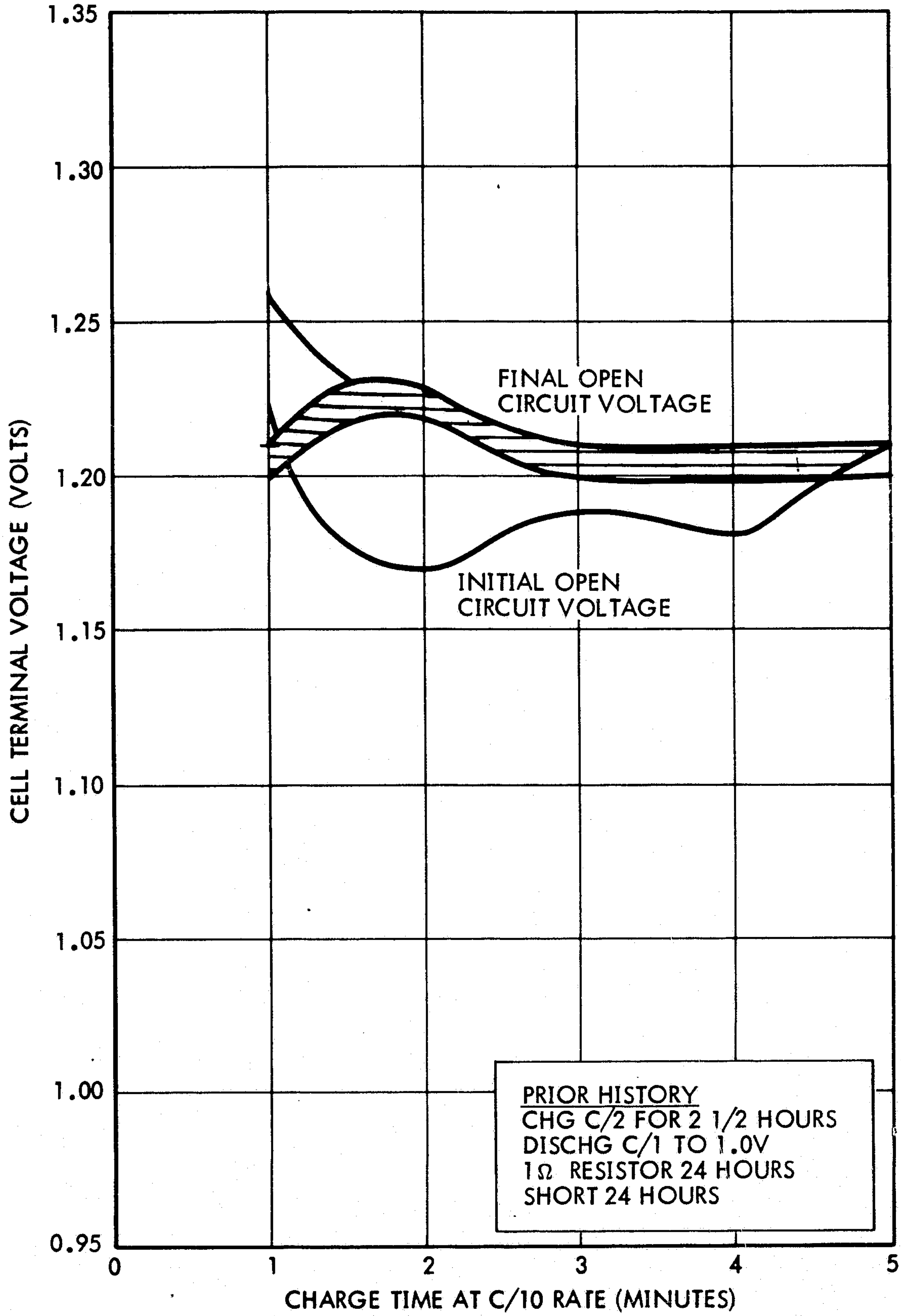


Figure 3. Voltage Decay Test Results, C/2 Conditioning, 24 Hour Short

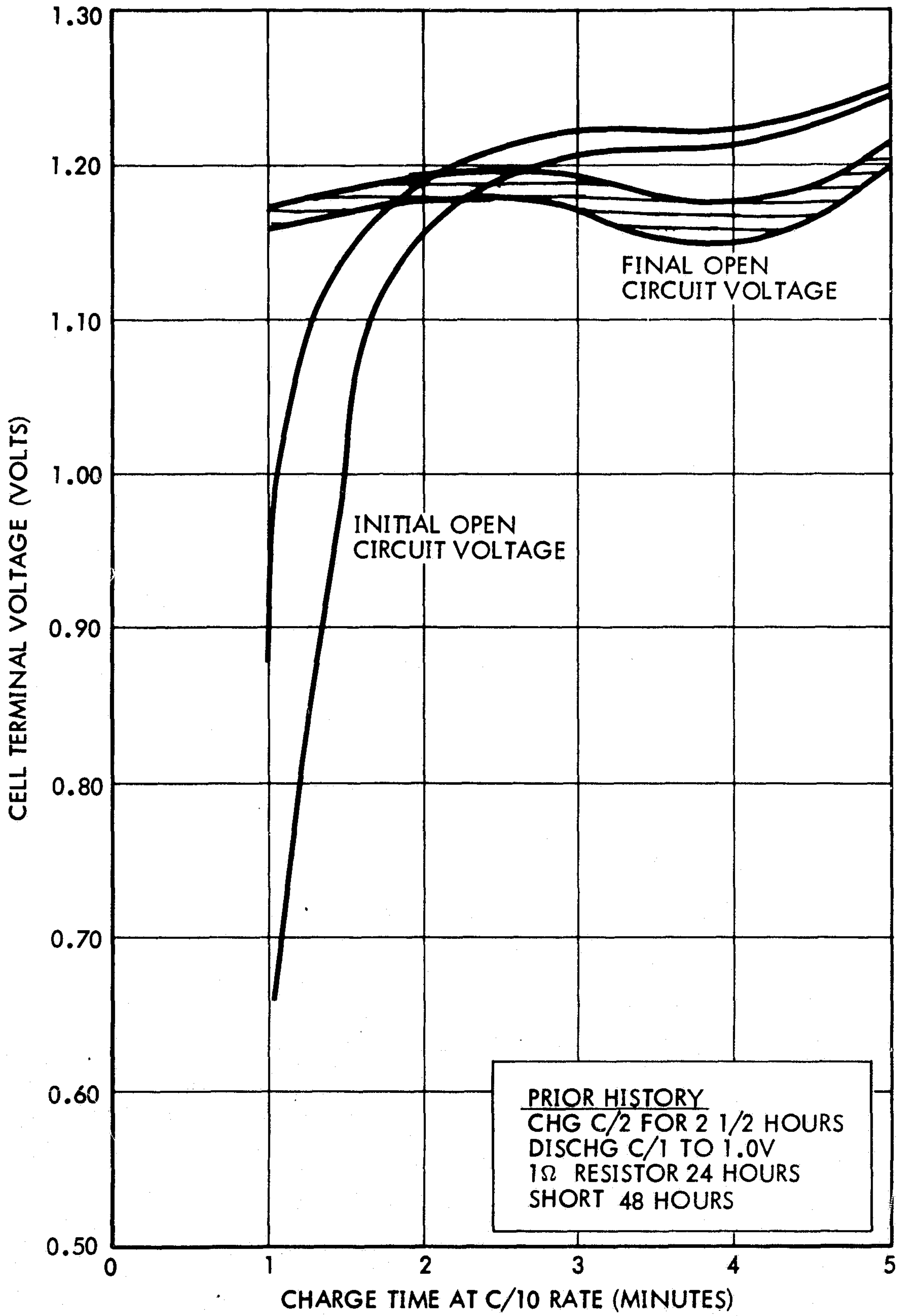


Figure 4. Voltage Decay Test Results, C/2 Conditioning, 48 Hour Short

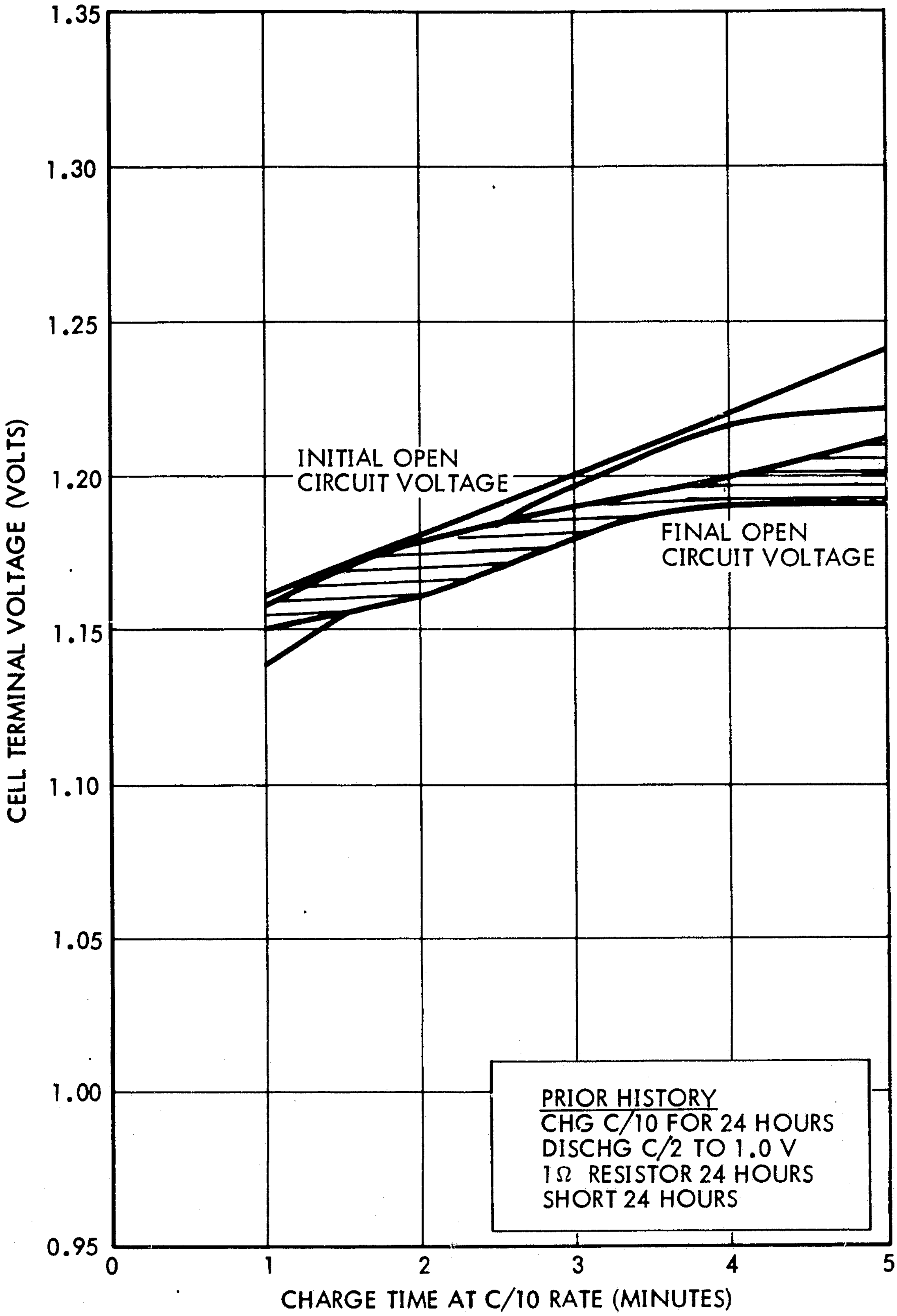


Figure 5. Voltage Decay Test Results, C/10 Conditioning, 24 Hour Short

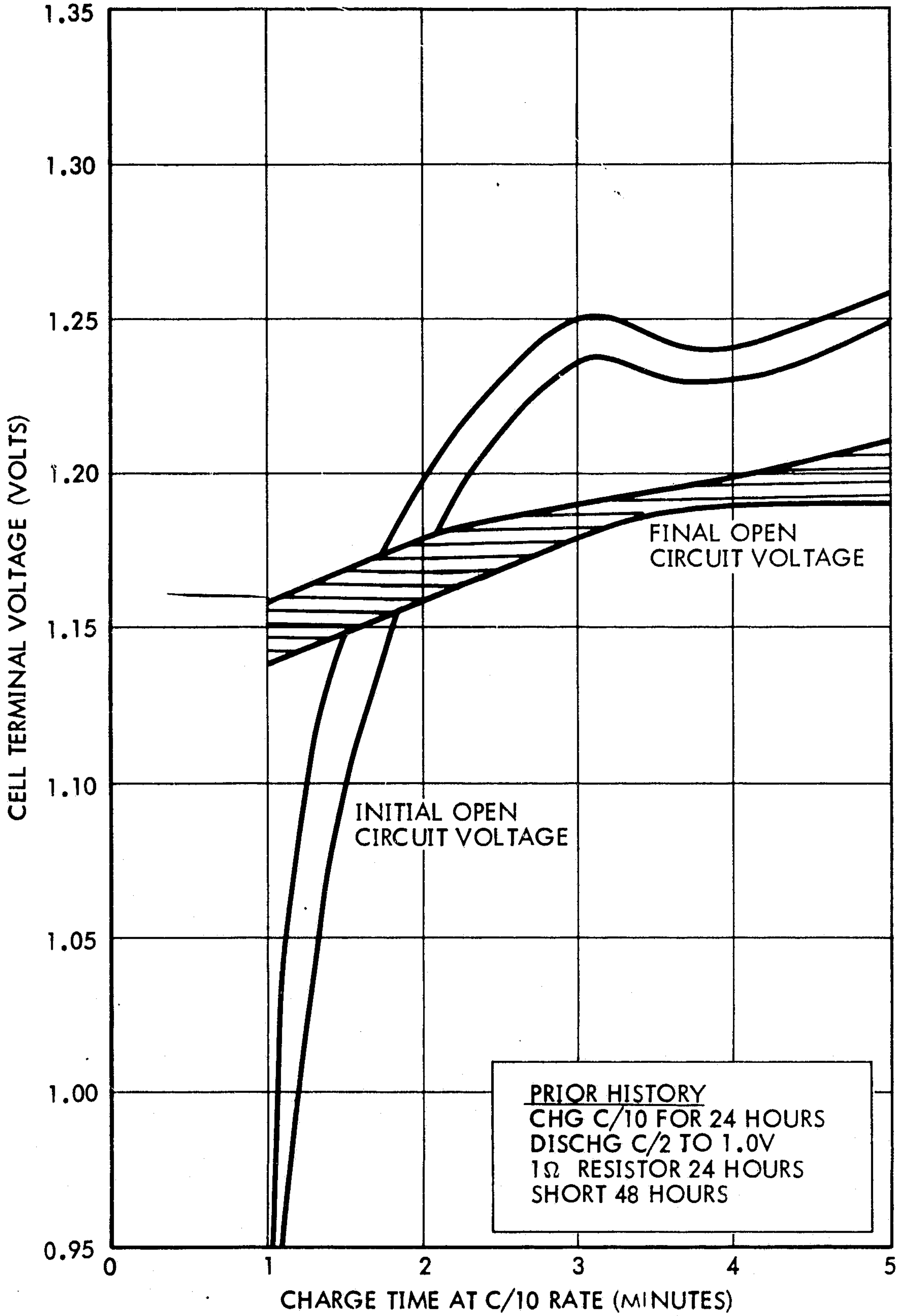


Figure 6. Voltage Decay Test Results C/10 Conditioning, 48 Hour Short

data, obtained by a test wherein charge acceptance was measured by discharging at the C/20 rate to 0.1 volt, show low charge acceptance at both charge rates, ranging from 18.2 to 31.3 percent at the C/10 rate, and from 14.6 to 37.2 percent at the C/2 rate. Although the average for the C/2 rate was slightly greater than that for the C/10 rate, the difference is considered not significant in view of the large scatter of the data. Thus there appeared to be no real advantage of using a charge rate higher than C/10 for the test charge. An in view of the fact that a C/10 charge rate is generally easier to implement and lends itself to a more accurate measurement of ampere-hours charged, the C/10 charge rate was used for most of the testing performed on the current study.

The other variable associated with the brief test charge used for the Voltage Decay Test is the number of ampere-hours injected. Historically most testing has been done using a 5 minute charge at the C/10 rate. This results in a throughput of approximately 1 percent of cell capacity.

A little information was available regarding ampere-hour inputs other than 1 percent. Thus Figures 1 through 6 each show data for five different inputs below 1 percent of cell capacity, obtained by charging for 1, 2, 3, 4, and 5 minutes at the C/10 rate. In sum, they show that there is little change in the results in the range from 3 to 5 minutes at C/10, but that with only 1 or 2 minutes charging the open-circuit voltage at 24 hours drops off sharply. Also at lower inputs the results appear more sensitive to other variables than when charging is continued for more than 3 minutes.

Data for inputs greater than 1 percent have not been published to the knowledge of the writer. Two sources of related data are available. Figure 7 (from Reference 4) shows a plot of the open-circuit potential of the nickel oxide electrode as a function of time on open circuit, for several charge inputs ranging from 0.33 to 5 percent of theoretical electrode capacity. First note that for a number of hours the potential decayed linearly with the largarithan of the stand time. At 0.33 percent charged this log-linear period was less than an hour; for 1 percent it was less than 24 hours, and for 2 and 5 percent it was greater than 48 hours. These results suggest that a 1 percent charge input may not be enough to assure a predictable positive electrode potential for

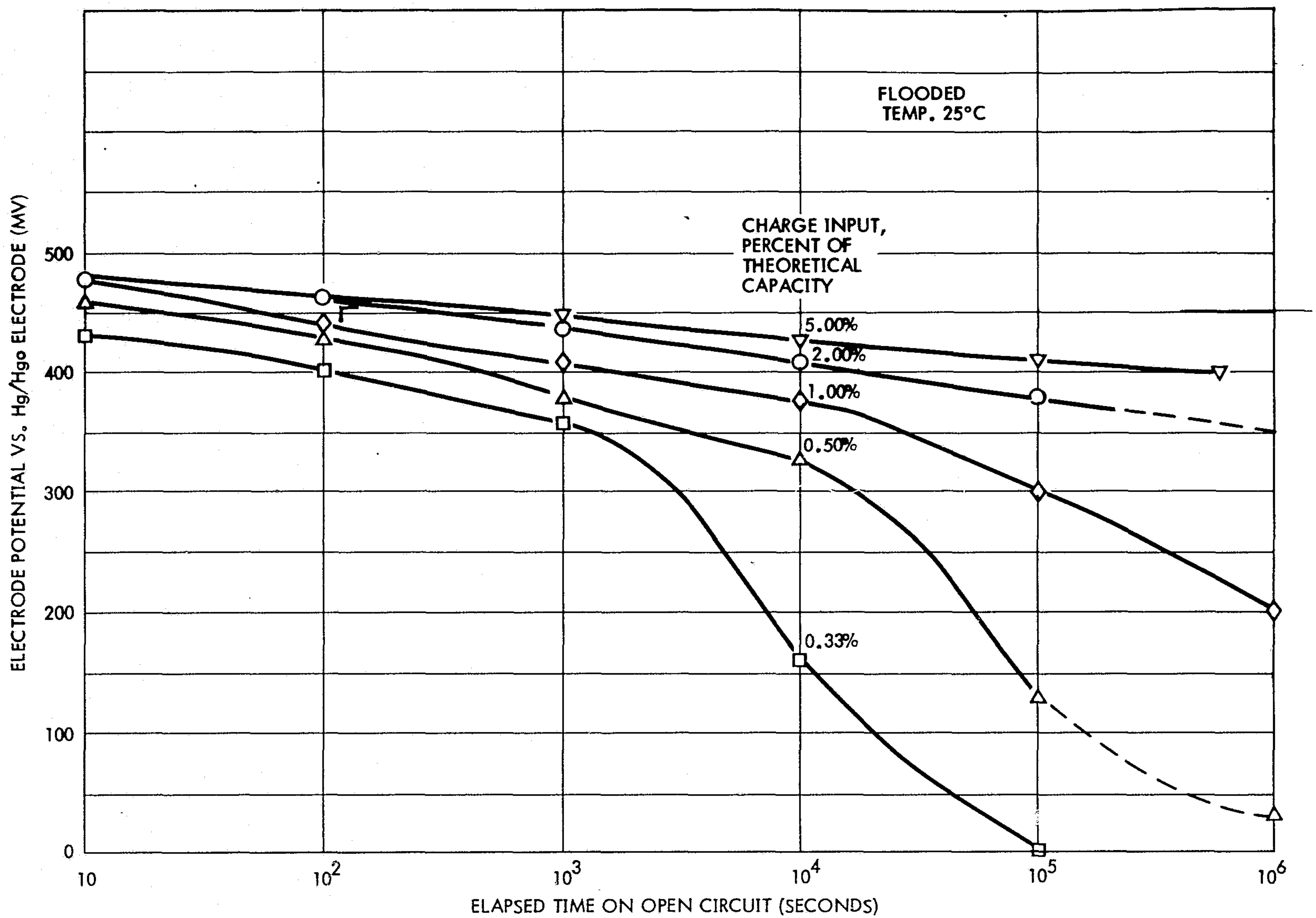


Figure 7. Nickel-Oxide Electrode Potential Decay Curves

24 hours even in the absence of an internal shorting path in a cell. On the other hand, an input of 2 percent appears to result in a much more predictable positive electrode potential over the time period of interest.

Data for charge acceptance for 1 and 2 percent ampere-hour inputs have been published (Reference 1). These data show no significant difference in charge acceptance between the two input levels, with the average for both (over 11 cells) being between 25 and 30 percent. However, because twice as many ampere hours were passed during the 2 percent charges, about twice as much charge is retained, a fact which should improve open circuit voltage stability. No open circuit voltage data was reported for short testing using a 2 percent throughput. Therefore, testing at charge inputs above 1 percent of capacity was included in the present study.

The temperature of the cells during the test is yet another variable that will affect the results, not only of the Voltage Decay Test, but of the other short tests or charge retention tests also. If the C/10 charge rate is used throughout, then charge efficiencies will change appreciably as the temperature changes. Temperature will have an even stronger effect on the rate of loss of charge of the positive electrode by chemical self-discharge on open circuit, where an increase in temperature will produce a more rapid loss rate and hence a more rapid decrease in cell voltage. Thus, for best results all cells subjected to this type of test must be kept at the same temperature, and the temperature should be the same for all cells subject to the same acceptance criteria. The specific effect of different temperatures was not investigated in this study. All tests were conducted with cells controlled at 22 ±3 °C.

In addition to the above, there is one less obvious test variable under the control of the tester that appears to be capable of affecting short test results; namely, the method used to restrain the cells during testing, and specifically the force applied to the sides of the cells. Considerable evidence indicates that the more the cell is compressed the poorer its short test and/or "charge retention" performance will be. This appears to be so even in cells in which there is no actual internal shorting path. The cause of this effect is not clearly understood. This

variable was not investigated during this study; instead, all testing was performed at the same value of force per unit area of plates in the cells.

A final test procedure parameter that can be changed by the user is the time allowed for the open circuit stand. The "standard" procedure calls for 24 hours; however, no information appears to be available as to whether or not this period is optimum.

If the open-circuit cell voltage changes continuously with time and in the same direction, then it would appear that a longer stand time would result in a more sensitive test (using a fixed voltage change criterion). Or additional reliability might be gained by taking readings at 24 and 48 hours, for example, and applying different but appropriate acceptance criteria to each. Thus there appears to be a large area for possible improvement of the test by extending the open circuit stand time beyond 24 hours. In this study stand times up to 100 hours were investigated.

3.2.2 Voltage Recovery Test Procedure Variations

The number of procedural variables that may affect the results of the Voltage Recovery Test is not as large as for the Voltage Decay Test as discussed above, because the number of steps are fewer. The variables that must be considered do include those listed as (a), (b), (c), and (d) under Section 3.2.1, as well as cell temperature and cell compression during testing as these aspects were discussed in the previous section. As the expected effects are the same for both tests, they do not require further comment here.

The effect of prolonging the open circuit stand time may not be the same as on the Voltage Decay Test, however. Whereas the voltage of cells on the Voltage Recovery Test usually rises during the first few hours of open circuit stand time, the subsequent direction of change of voltage may not always be increasing, even in a non-shorted cell. The open circuit voltage will continue to increase only as long as there continues to be some minimum quantity of charged positive (and negative) active material. If the positive electrode becomes depeleted during the stand time through self-discharge (either because of an insufficient state of charge at the start or a rapid rate of self-discharge), its potential would begin to change in the direction of that of the negative electrode.

Under these conditions, where depolarization tends to increase cell voltage and a depleted electrode tends to decrease cell voltage, the cell voltage would not necessarily be indicative of an internal short.

The Voltage Recovery Test procedure as specified in Reference 6 (Section 6.4.2) calls for a 16 hour minimum discharge on one ohm following a charge and a discharge, immediately prior to the open circuit stand. If only a 16 hour period on the resistor is used, it is likely that the positive would have enough residual charge to support the test for 24 hours. However, the sensitivity of the test is likely to be low under these conditions. If, on the other hand, the voltage is observed over a stand period longer than 24 hours, better results, particularly for marginal cells, might be obtained. Conversely, if the resistive discharge time is made longer than 16 hours, the subsequent stand period might be shortened with no loss in sensitivity. A limited investigation of these relationships was included in the experimental part of this study.

3.2.3 Charged Stand Test Procedure Variations

The Charged Stand Test is subject to fewer procedural variables under the control of the tester than the other two tests. The actual number depends on which steps are considered an integral part of the test, and which are considered as pre-test conditioning. Inasmuch as immediate prior history and/or conditioning may significantly affect the capacity measured prior to the charge preceding the open circuit stand, it would appear advisable to include definition of at least one prior cycle in the procedure per se. If this is done, then whether or not the cell is resistively discharged to a low voltage prior to the calibration cycle becomes an important test variable, as the capacity measured following a "short-down" is usually higher than that measured on the second cycle without shorting down in between. The comments above regarding the effects of cell temperature and cell compression apply here also. The discharge rate used for capacity measurement before and after the open circuit stand should have only a minor effect on the results, assuming that the same rate is used each time, and that the same end voltage is used for the measurement.

One source of variability that needs more attention is the amount of open circuit stand time allowed between the end of the charge and the beginning of the discharge used to measure capacity at the beginning of the test. Different testers use different times, ranging from zero to several hours. Because the cell is overcharged appreciably prior to the discharge (typically 2.4 C ampere-hours are charged at the C/10 rate), a certain amount of what may be termed "overcapacity" is created. This overcapacity largely disappears by spontaneous self-discharge of the positive electrode within a few hours after charging is stopped if the cell is left on open circuit. If the cell is discharged immediately after the end of charge, much of the overcapacity is included in the capacity measured. Then, when in the course of the Charged Stand Test, the cell is again charged, overcharged, and put onto open circuit stand, the loss of overcapacity is included in the difference calculated between the initial and final capacity values. If the overcapacity produced were controllable and reproducible, it could be deducted from the capacity difference, thus giving a net change that would better represent the long-term retention capability of the cell. However, the overcapacity is probably variable, and may be large enough to seriously interfere with the main purpose of the test.

In view of the above, it is suggested that some open-circuit time should be allowed between the charge and the discharge of the initial capacity cycle. No effort was made to determine an optimum stand time for this purpose during this study; a time of 1 hour was chosen as probably adequate after a C/10 charge, and all tests were made using this same open circuit stand time before the discharge.

4. EXPERIMENTAL PROGRAM

4.1 General Approach

The following approach was used for the testing and data acquisition performed under this study.

a) All physical and environmental conditions were carefully controlled throughout testing.

b) All electrical inputs and outputs were carefully controlled and precisely measured.

c) Voltages were measured at relatively frequent intervals, varying from once each twenty minutes to once every four hours during the "open circuit" periods (instead of measuring voltage only at the end of an open circuit stand period).

d) Open circuit voltages were plotted versus open circuit stand time and, in most cases versus the logarithm of the open circuit stand time. The entire curves were used to characterize the response.

It might be argued that the taking of many voltage readings over a 24-hour period does not represent true open-circuit conditions, and hence would prejudice the results. In answer, it is pointed out that previous (unpublished) experience indicates that cells without internal shorts give essentially identical response over a 24-hour period with a 100 K-ohm resistor across the terminals as they do with no resistor attached. In the data taking arrangement used for the present study, the effective load placed across the cell during a direct measurement was approximately 2 megohms, the average dwell time per measurement was 5 milliseconds per cell, and the maximum number of measurements per cell per 24-hours during open circuit periods was thirty. Thus the "loading" of the cells by the data acquisition system was completely negligible and could have had no observable effect on the results.

The idea of plotting the voltage as a function of the logarithm of the stand time is based on the following argument. As shown in Figure 7 (from Reference 4), the electrode potential of a partially charged nickel oxide electrode decays in proportion to the logarithm of the open circuit stand time for some period of time. The time span over which the potential is log time linear increases as the amount of charge increases. Therefore, as the potential of a partially charged cadmium electrode normally changes very little over the time-span of interest, it was reasoned that the open-circuit terminal voltage of a nickel-cadmium cell having no internal shorting paths should show the same change with time as does the positive electrode potential. Also, it was hoped that the effect of an internal short would be to cause the cell voltage curve to be

non-linear with log time and thus allow an internal short to be detected by the shape of the curve independent of any absolute voltage criteria.

This approach was shown to be promising by reference to some recently obtained multiple voltage data taken during a 7-day charge stand test. Figure 8 (from Reference 7) shows the data for several cells plotted on a linear time scale. Figure 9 (from Reference 7) shows data for one of those cells plotted versus log time in hours. The fit to a linear relationship appears excellent. It was recognized that the data shown was for a high state of charge of the positive electrode, so the applicability of this type of plot to very low state of charge remained to be demonstrated.

4.2 Test Program

4.2.1 Scope of Testing

The testing reported was performed over a 4-month period extending from August through December of 1974. No new cells were purchased for the purpose of testing on this project, and hence all the cells tested were those already in-house at TRW Systems. Therefore, the choice of cells to test was limited to those available with the associated history at the time the work began.

The types and ranges of variables included in the test program are summarized below:

Variable	Range Tested
Shorted Storage Time	2 months to 4 years
Conditioning (One Cycle)	Yes and No
Post-discharge Resistor	6/C to 15/C ohms
Post-discharge let-down time	16 to 40 hours
Shorting after resistive discharge	Yes and No
Charge rate for brief charge	C/10 and C/2
Throughput during brief charge	0.5 to 3% of cell capacity

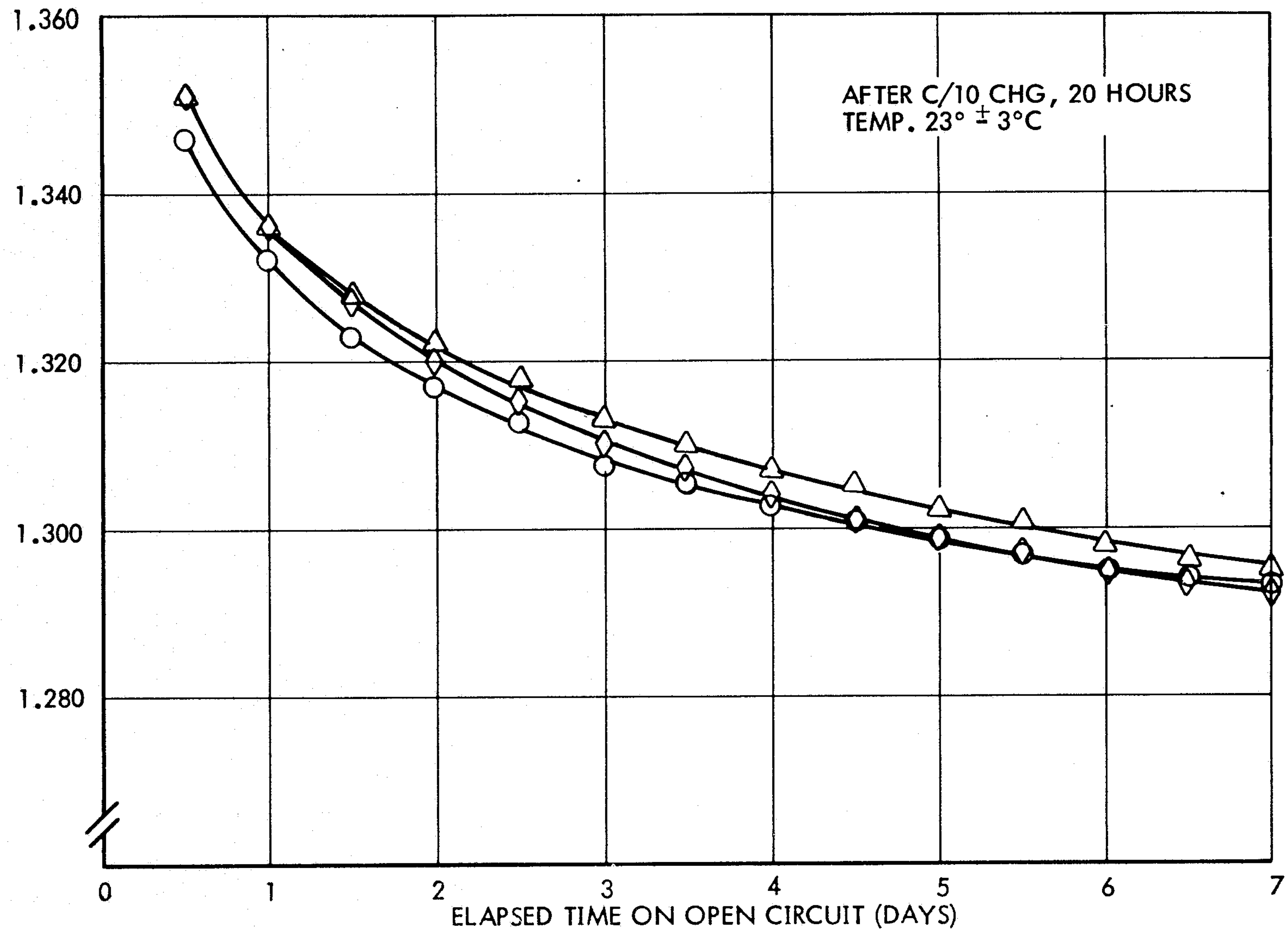


Figure 8. Charged Open-Circuit Stand Data - Linear Time Scale

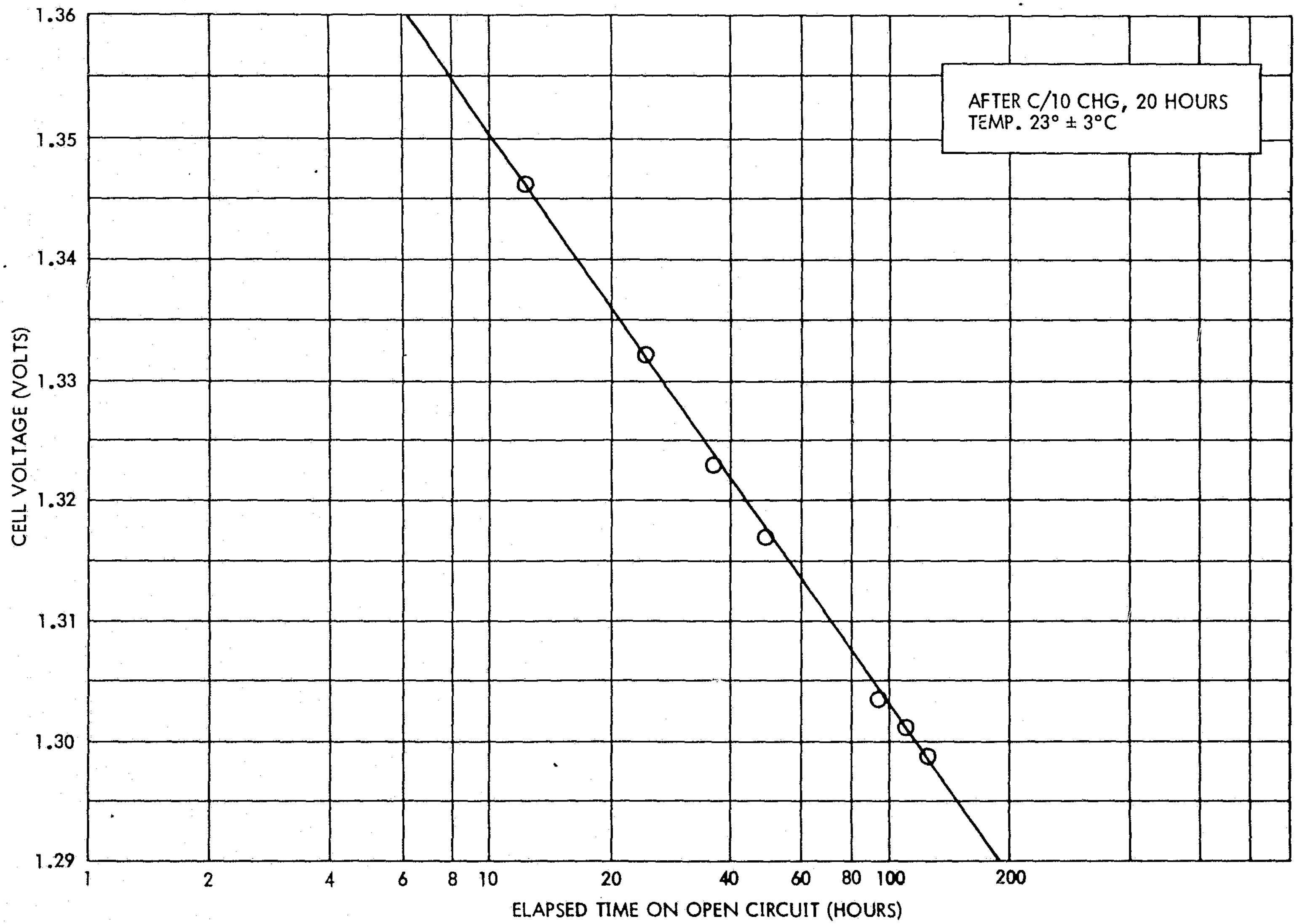


Figure 9. Charged Open-Circuit Stand Data - Log Time Scale

Not all cells were tested with all variables, and no one set of cells were subjected to all combinations of variables. The same variables were used with several groups of cells, particularly with cells from different manufacturers and with cells in different age groups. The test program is described in more detail below.

4.2.2 Test Articles and Test Procedures

Cells tested ranged in capacity from 12 to 50 ampere-hours. Most of the cells tested had nylon separators but some of the 50 Ah cells tested had polypropylene separators. All of the older cells tested had been stored in the discharged and shorted condition. Cells from two sources were included: General Electric, Battery Products Section; and Gulton Industries, Alkaline Battery Division.*

In general, five test sequences were performed, each sequence involving a different lot of cells. These test lots are identified in Table 4-1. The testing was performed in accordance with written test procedures, copies of which are included as Appendix A to this report.

Table 4-1. Cell Test Lot Identification

Test Sequence and Lot Number	Cell Manufacturer	Cell Capacity (AH)	Number of Cells in Test Lot	Age and Other Data
1	General Electric	24	48	Two months
2	Gulton	12	14	Two years; on shorted storage 1 year
3	Gulton	15	54	Five years; on shorted storage 4 years
4	General Electric	12	12	Five years; on shorted storage 4 years
5	General Electric	50	48	Six months; 24 units with nylon; 24 with polypropylene

*Now SAFT America

All data was acquired by an automatic data system and stored in digital format on magnetic tape. During the tests, sets of data were printed out on hard copy at prescribed time intervals. Details of scheduling for data acquisition are contained in the test procedures.

4.3 Test Results

In the following sections the results obtained from the different test sequences are presented in a factual manner with a minimum of comment. The data shown has been selected to be representative and to present significant information. More data than that shown was acquired but is not plotted because it is very similar. These results are discussed in Part 5 of this report.

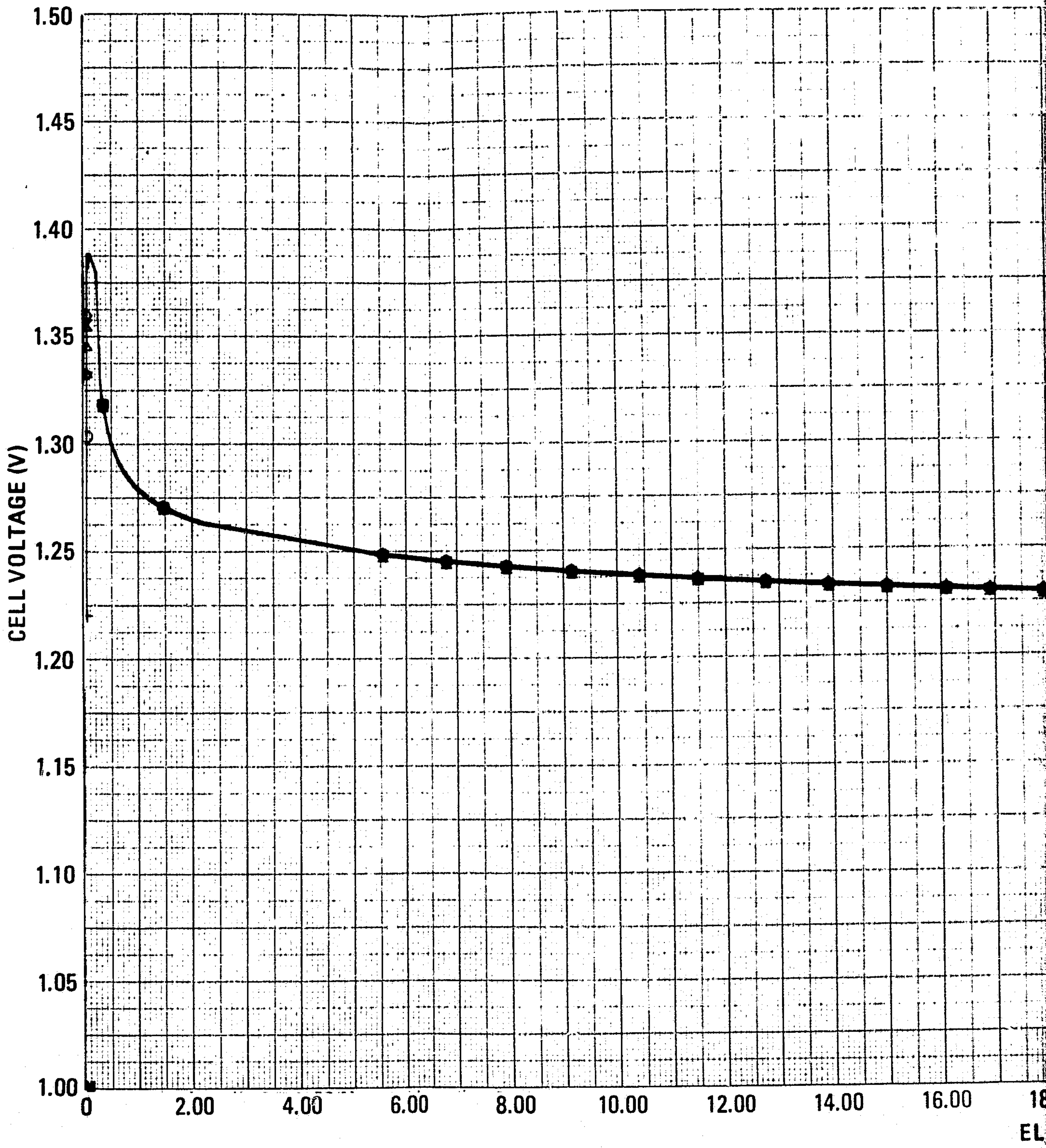
4.3.1 Results From Test Sequence No. 1

The first test of Sequence No. 1, involving new 24 Ah cells, was an open circuit stand immediately following removal of shorts. The cells had been shorted continuously for several weeks previously. No cycling was interposed. The range of cell voltages at the end of 24 hours on open circuit was 0.186 to 0.230 volt on this test.

Various cells in Group 1 of Lot 1 were then given various brief charges and put on open circuit. Group 2 was first given a full charge-discharge cycle and then the cells were given the same charges as Group 1 before being put on open circuit. A plot of the voltages of the six cells in Group 1B on a linear time scale is shown in Figure 10. Plots of the other groups appeared similar. Note the small spread of voltages even after 40 hours.

Plots of open circuit voltages versus log open circuit stand time for Group 1B and two other groups are shown in Figure 11. Note that the points for most of these groups appear to lie along reasonably straight lines from 4 hours to beyond 48 hours when plotted in this manner. In these plots a single data point indicates that the spread of the six voltages in a group was less than 10 millivolts, and the point is located at the mean value of the voltages.

It may be seen that the effect of increasing the charge input from 6 minutes (curve 1) to 12 minutes (curve 2) at C/10 was to raise the



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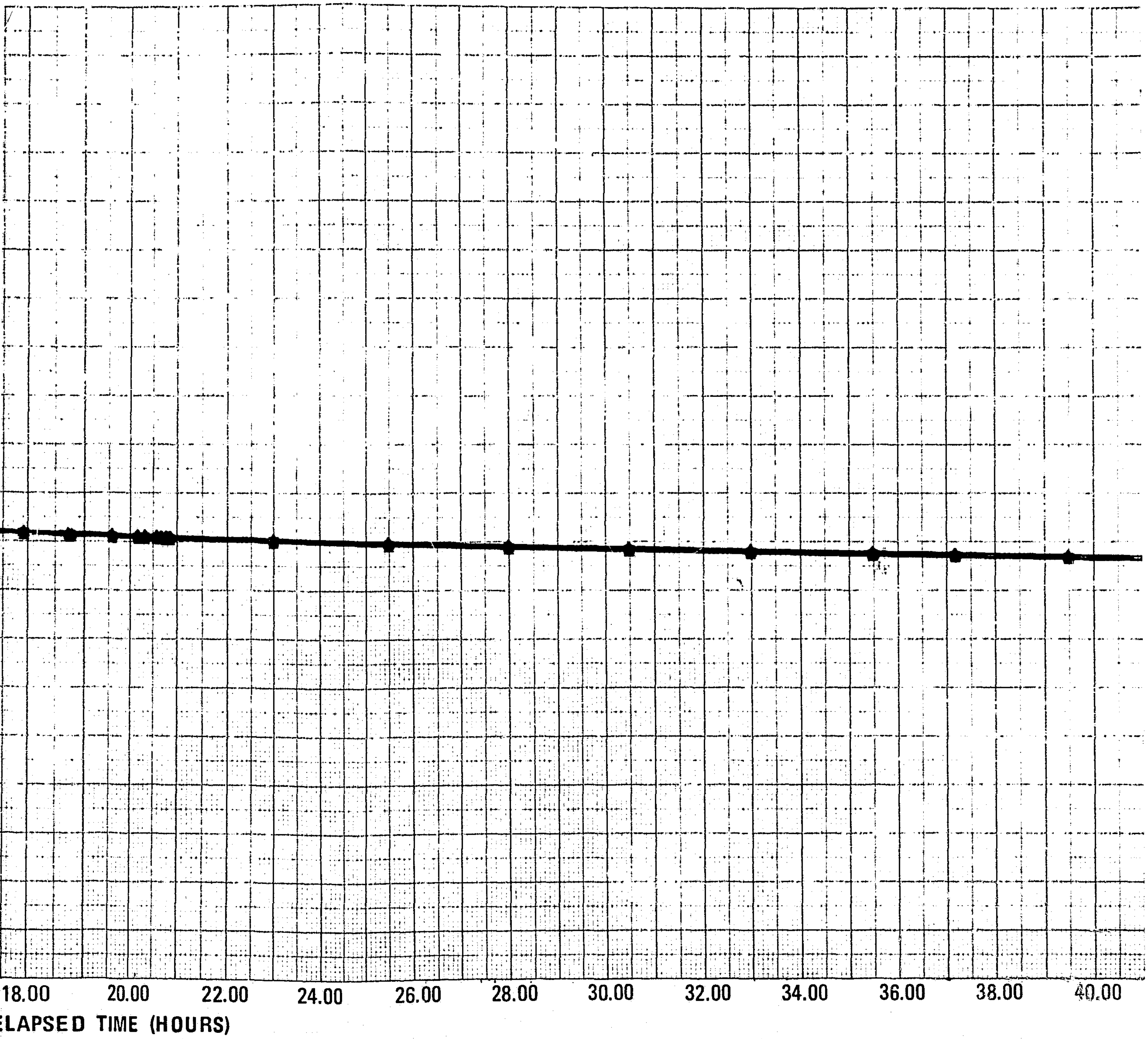

Figure 10. Voltage Decay Data, Test Sequence No. 1, Subgroup 1B

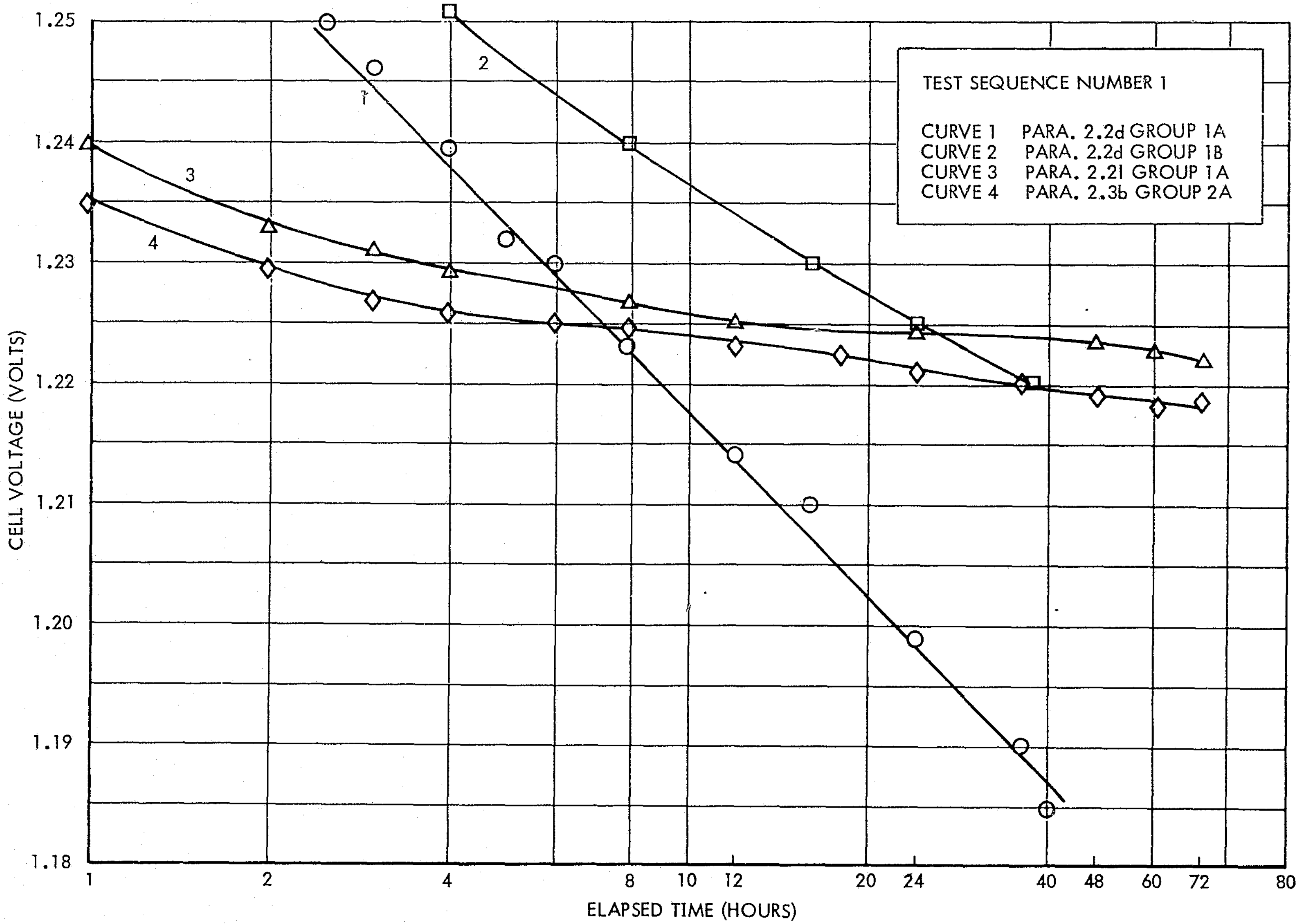

Figure 11. Semi-log Plot of Data From Figure 10

voltage level over the entire curve (by 26 mV at 24 hours). The actual voltages at 24 hours were 1.199 for Group 1A and 1.225 for Group 1B.

A set of voltage curves for Group 1C cells, all of which had resistors attached, is shown in Figure 12, for the first Voltage Decay test made on these cells (prior to cycling). A group charged for 12 minutes was selected for this initial testing with resistors, instead of cells charged for 6 minutes, because at the time it appeared that a 6 minute charge might be insufficient to give good results. Note that a different vertical scale is used in Figure 12 for the cells with 100 ohm resistors in order to show the voltages below 1 volt. This different scale makes the voltages of these cells appear greater than those of the other cells early in the test, whereas in reality voltages of cells with the 100 ohms attached were always lower than the rest.

The voltage behavior of the Group 1A cells on the second Voltage Decay Test (Test Sequence No. 1, para. 2.2(1)) following a single charge-discharge cycle, is shown by Curve 3 in Figure 10A. The (negative) slope was much less than when tested before the conditioning cycle (Curve 1) yet the voltages after 8 hours were higher.

Group 2 cells in Test Sequence No. 1 were given a charge-discharge cycle before the first Voltage Decay Test. A plot of voltage data on a linear time scale from the first Voltage Decay Test on Group 2A (no added resistors) is shown in Figure 13. Note the trace for one anomolous cell which dipped below the average of the other five cells by 25 mV soon after opening the circuit, then returned to join the others after 12 hours. A semi-log plot of the data for Group 2A for the first Voltage Decay Test is shown as Curve 1 in Figure 14. This same data is plotted on Figure 11 as Curve 4. Voltages at the same stand time were close to those for the second test of Group 1A, as can be seen by comparing curves 3A and 4A in Figure 11.

Figure 15 shows the data for Group 2C where resistors were attached (Test Sequence 1, para. 2.3(f)). As in Figure 12, the scale for the cells with 100 ohm resistors is different from that for the other cells. With Group 2, as with Group 1, the response of cells with 10K ohm resistors attached cannot be distinguished from that of cells with no external resistors even after 72 hours of stand time.

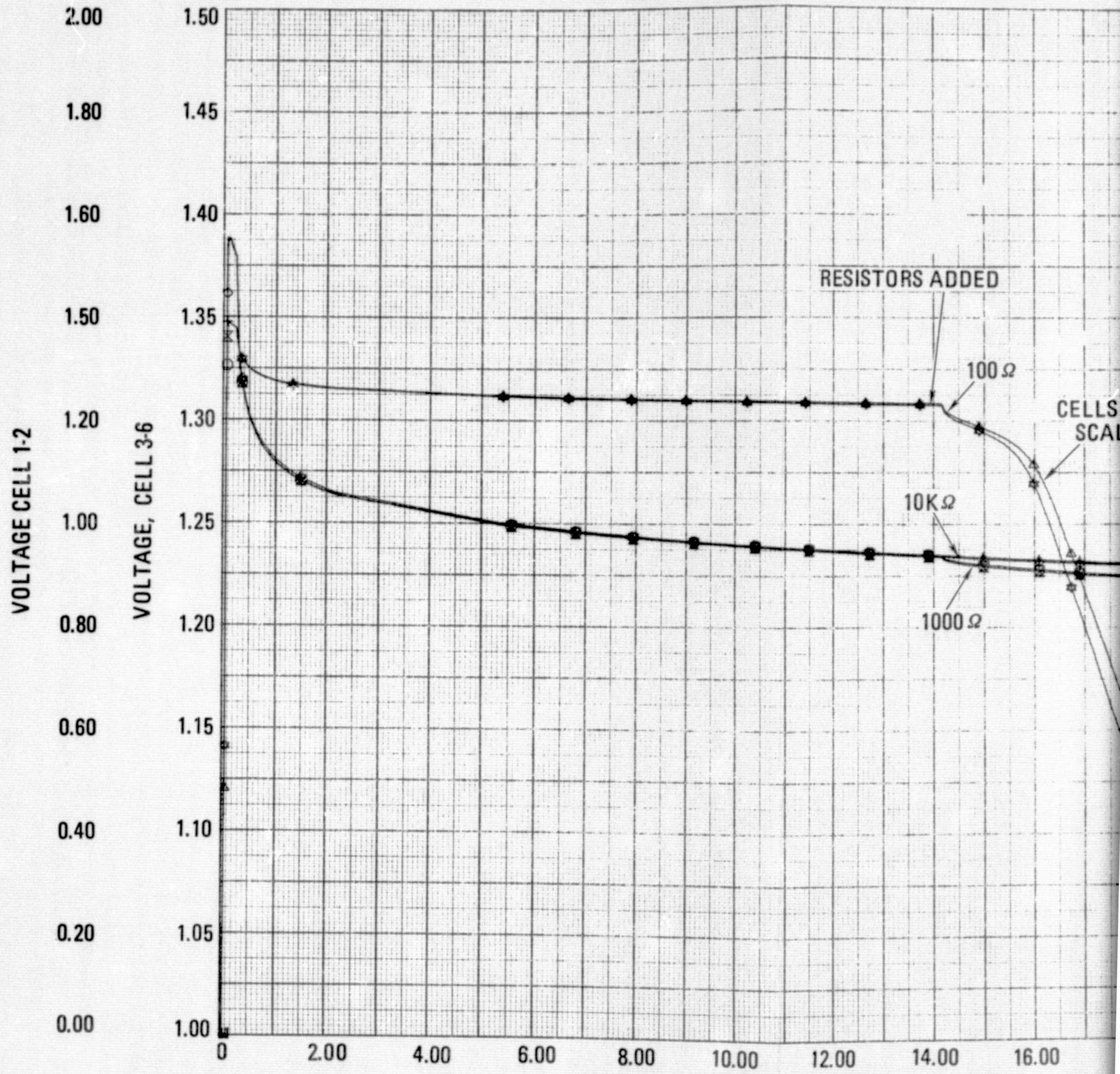

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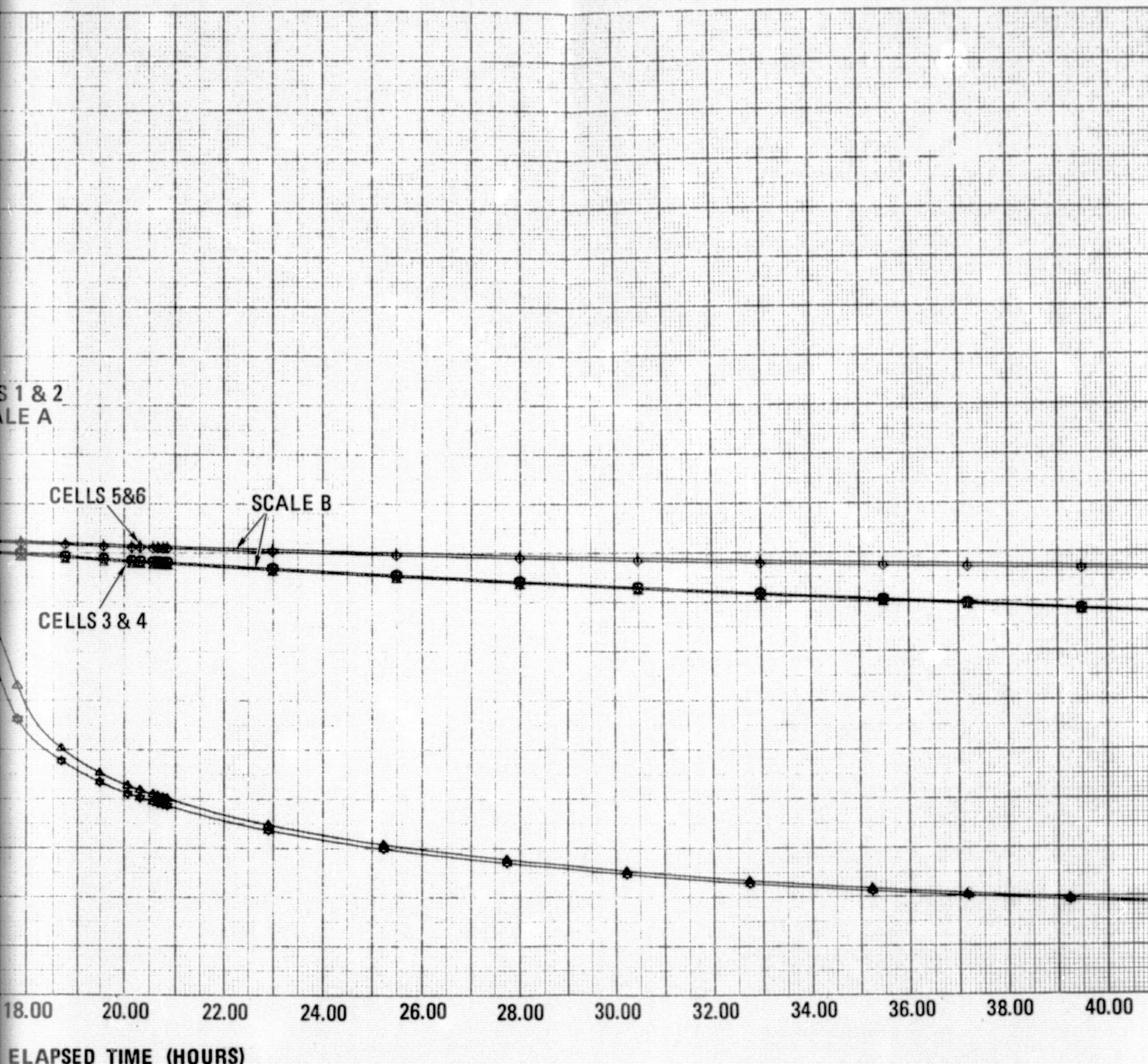
Figure 12. Voltage Decay Data, Test Sequence No. 1, Subgroup



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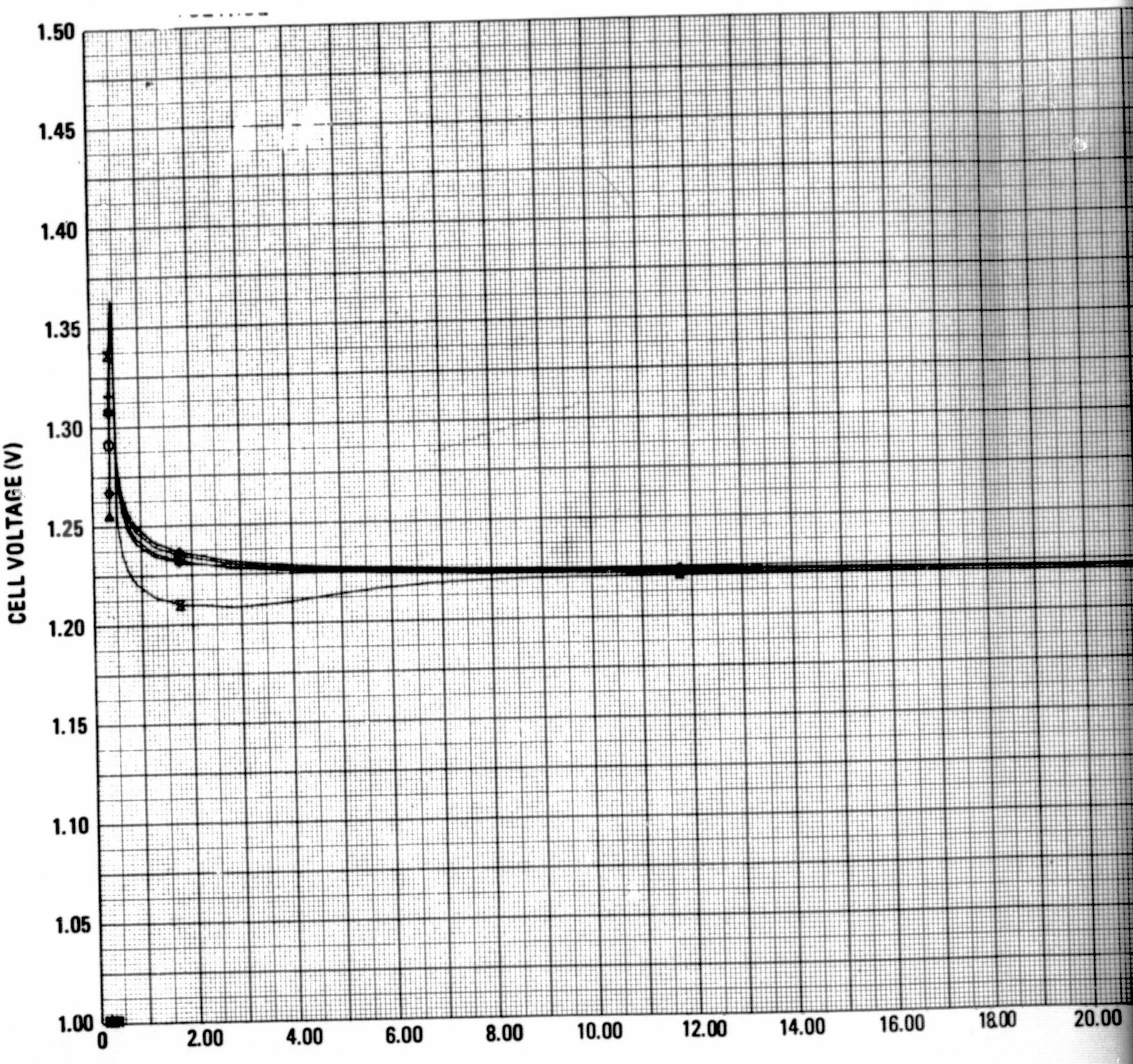

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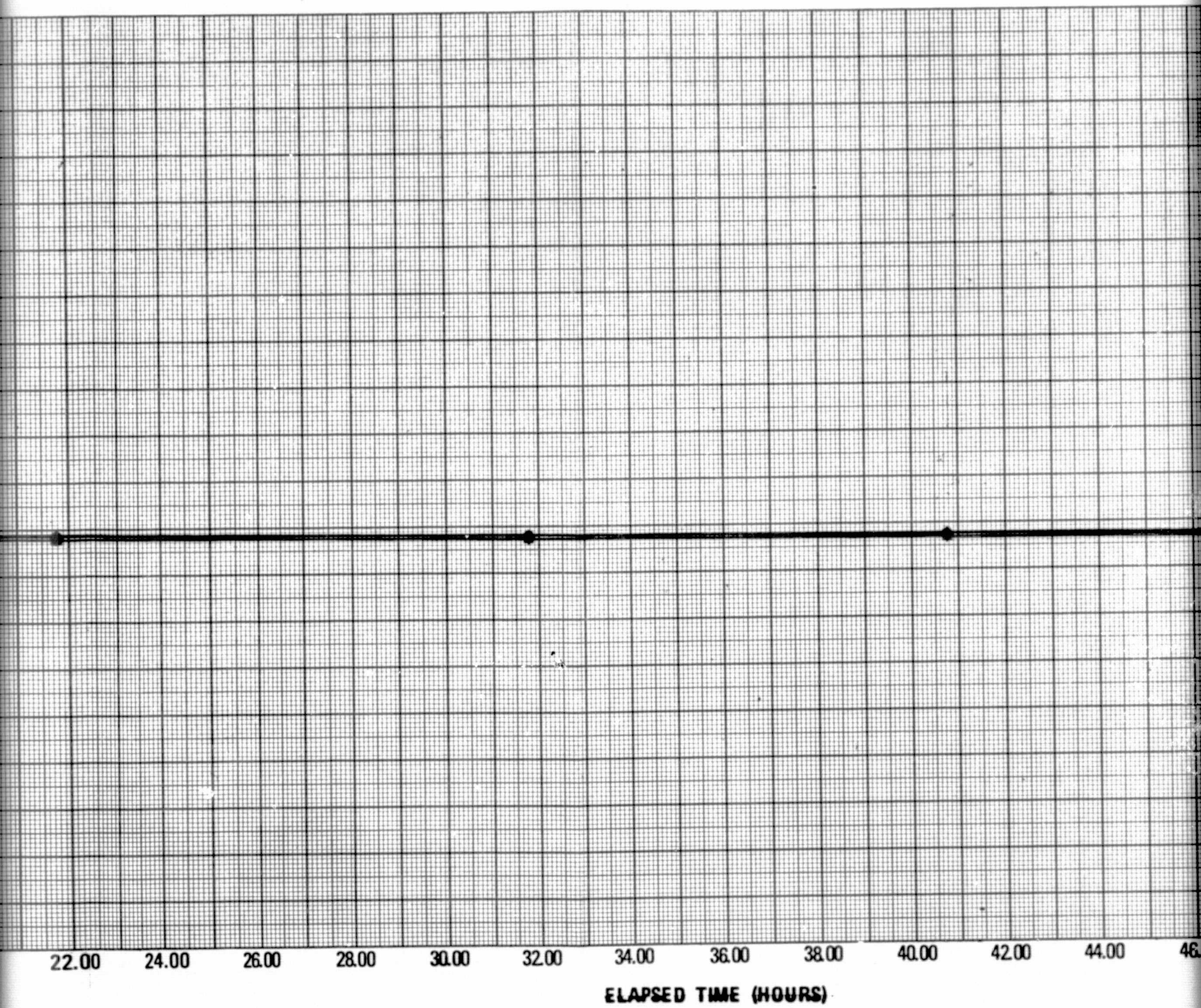

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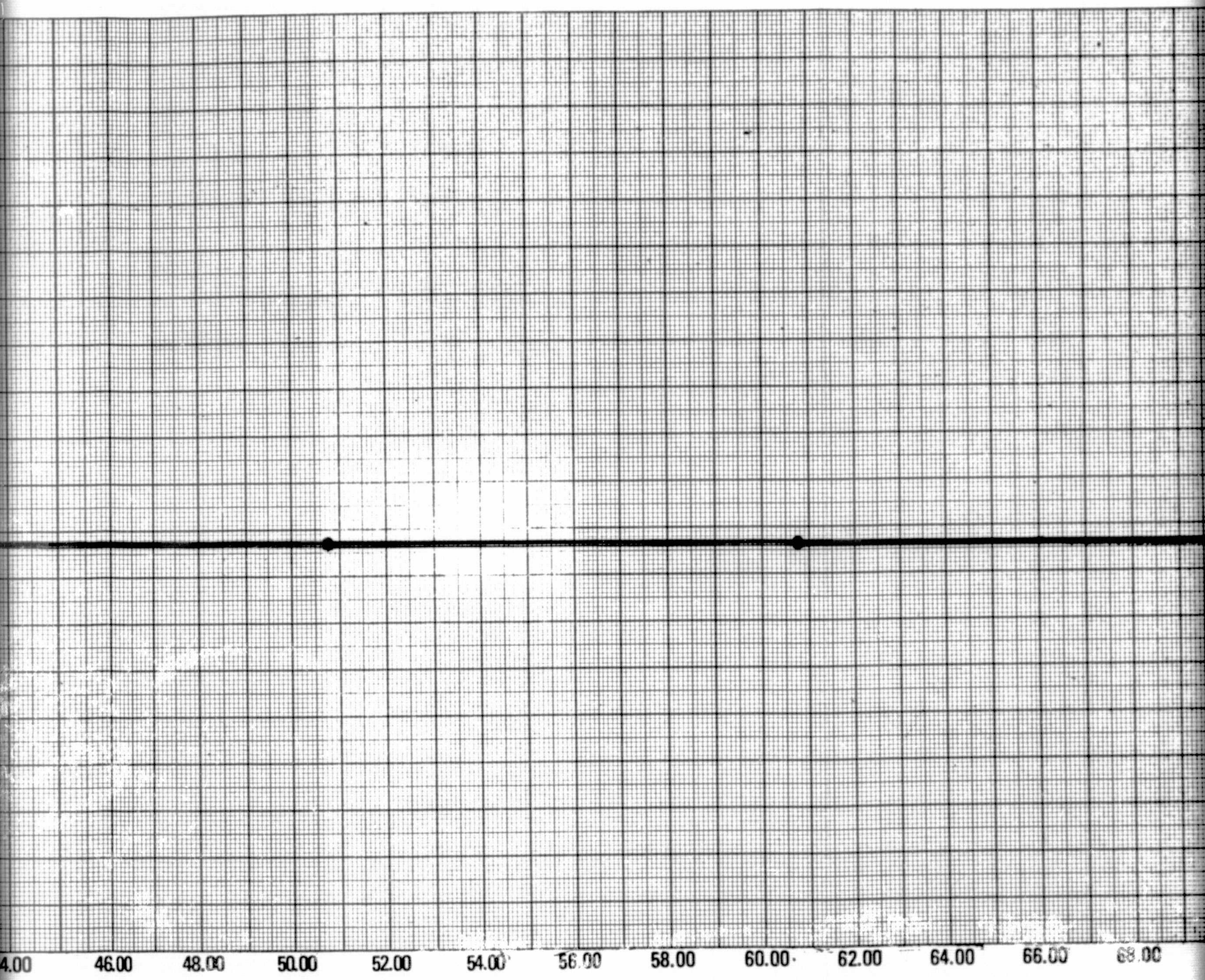

Figure 13. Vol...ge D...
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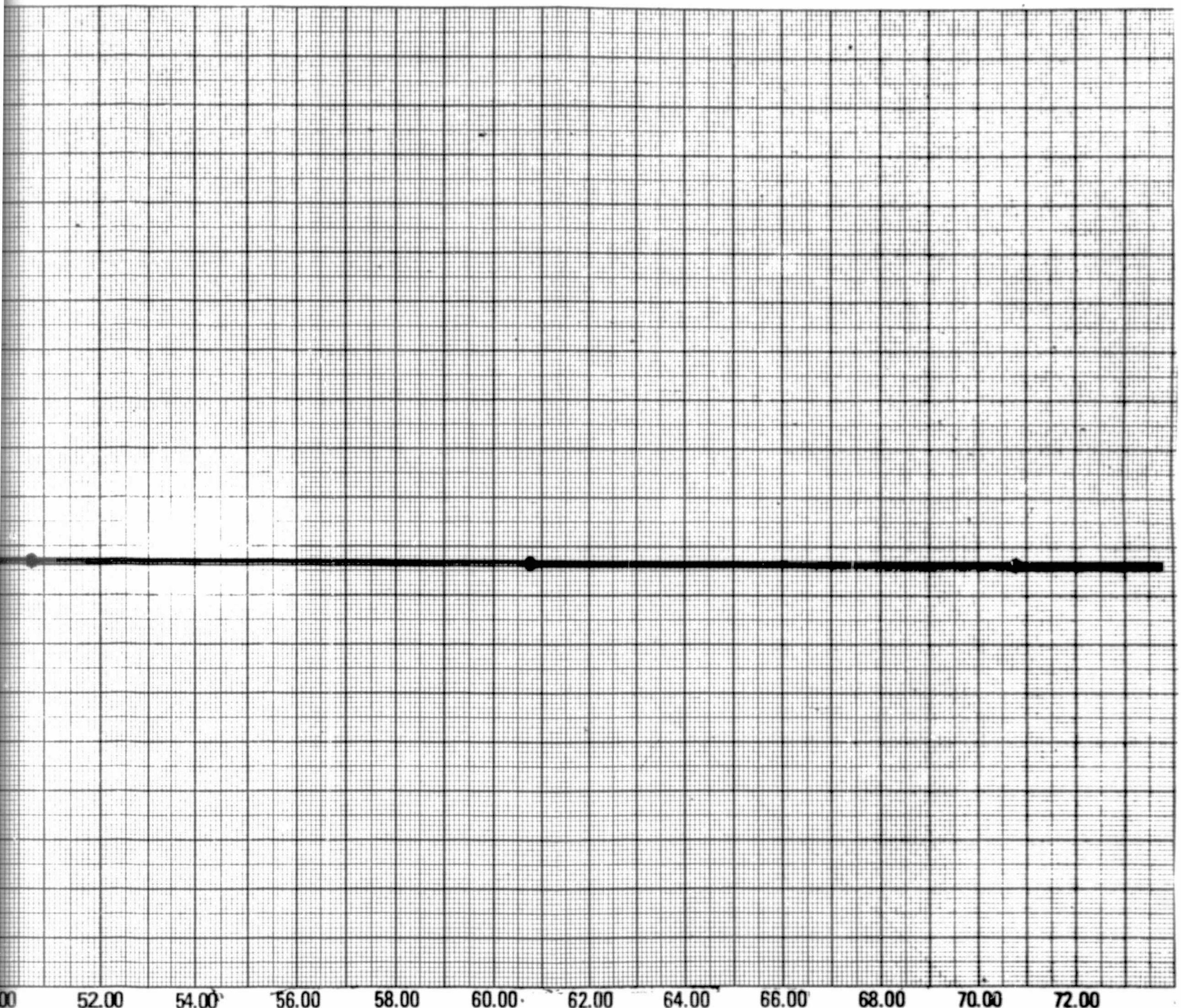

Figure 13. Voltage Decay Data, Test Sequence No. 1, Subgroup 2A

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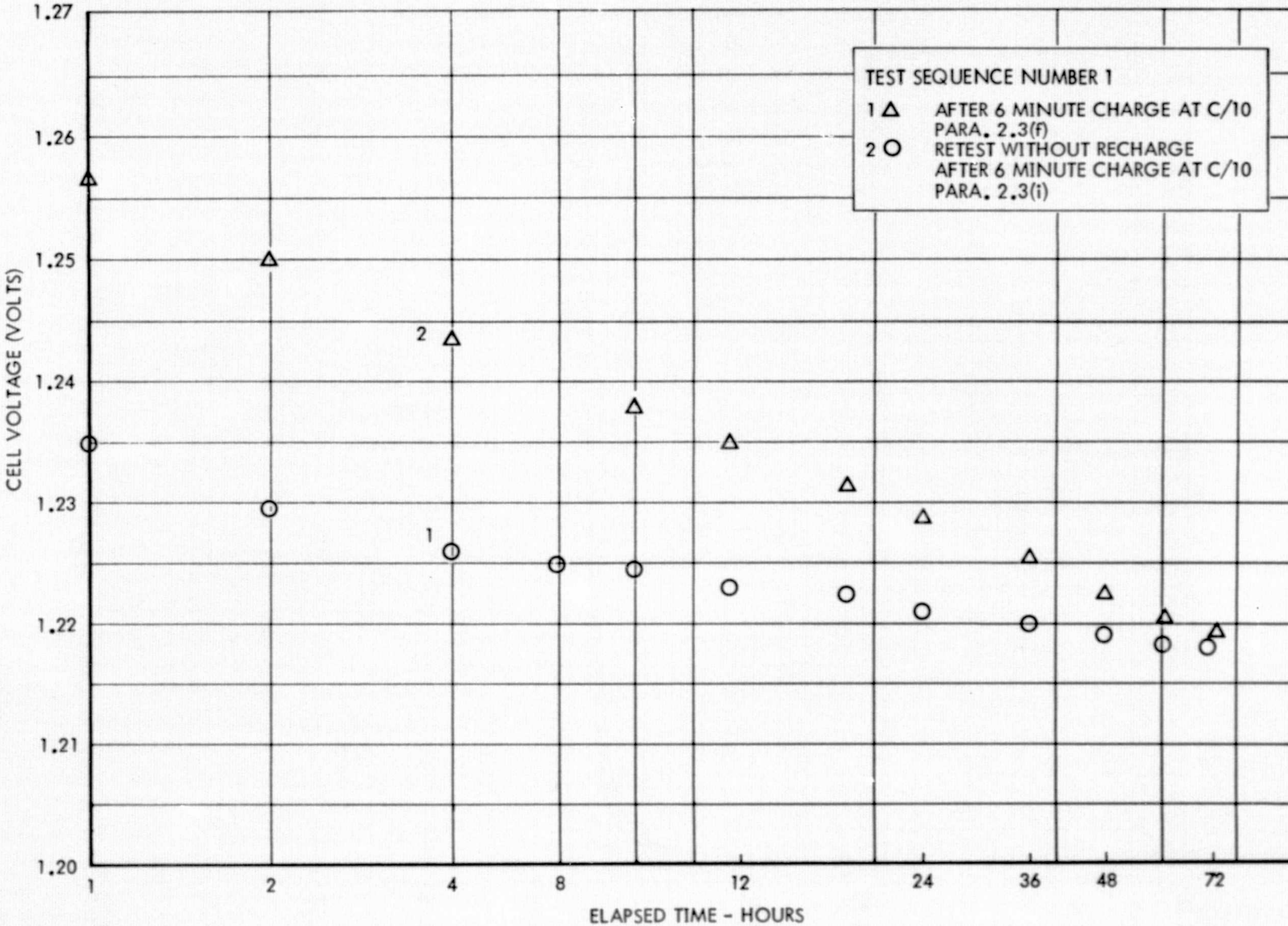
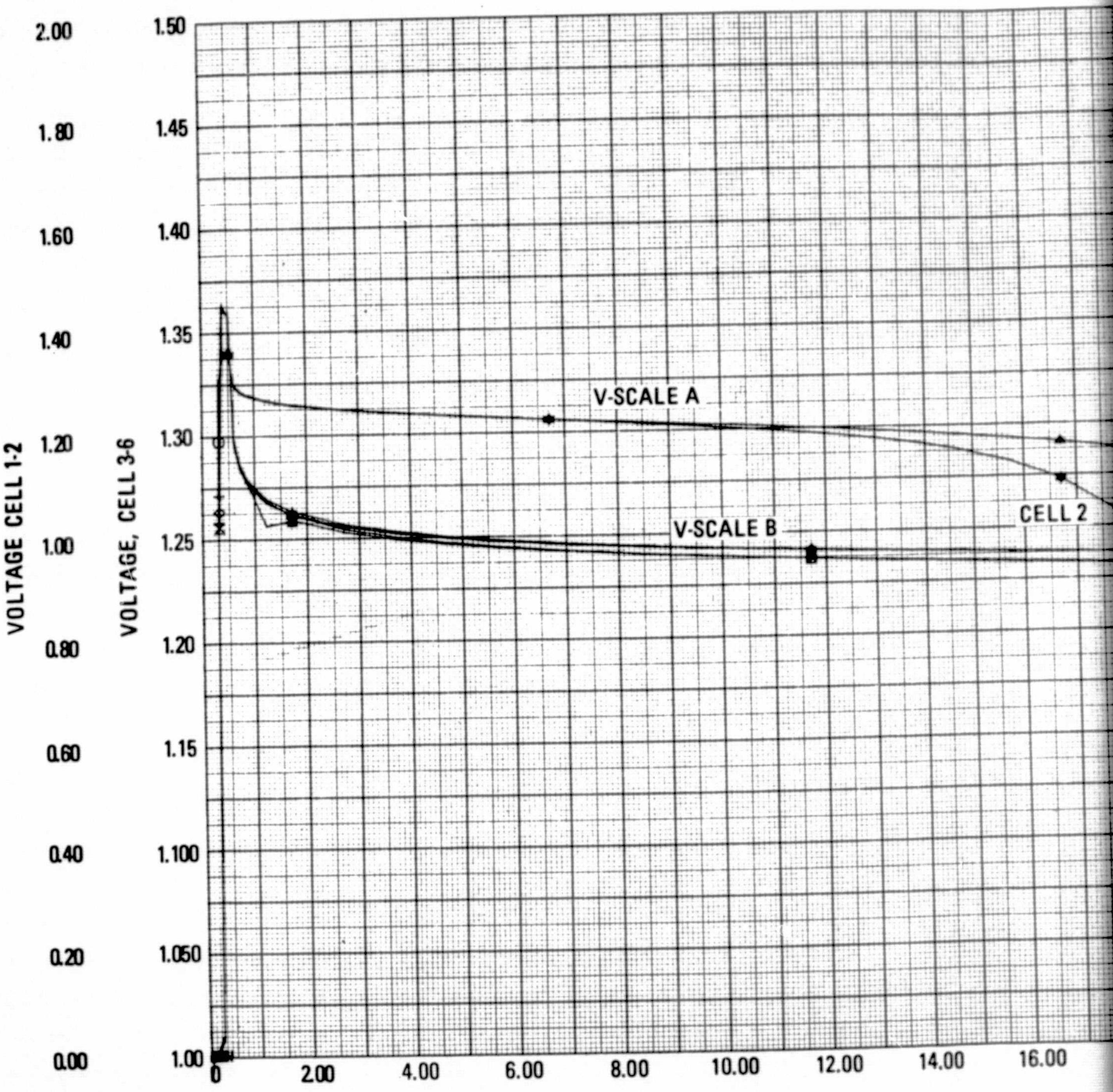


Figure 14. Semi-log Plot of Data From Figure 13



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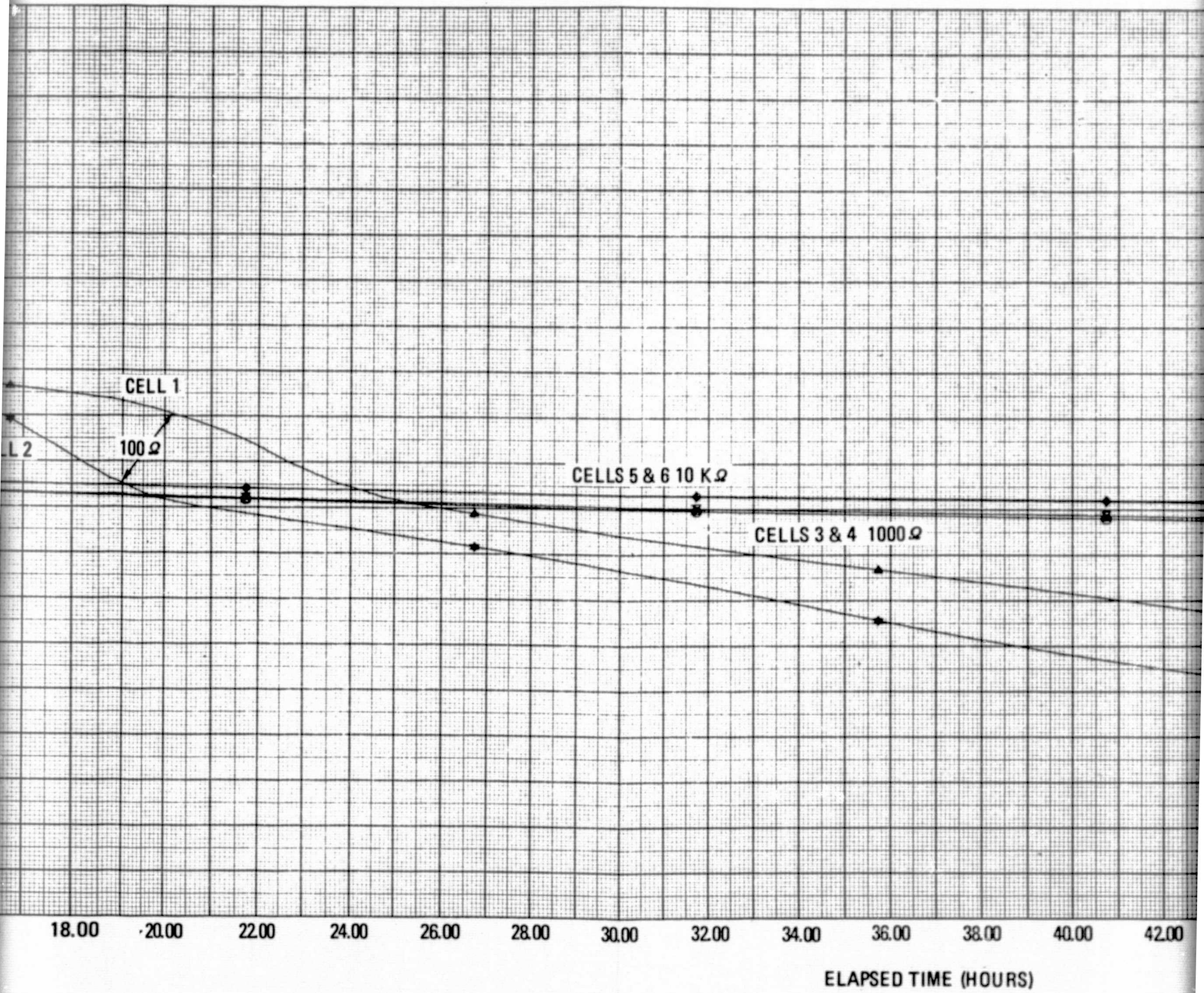

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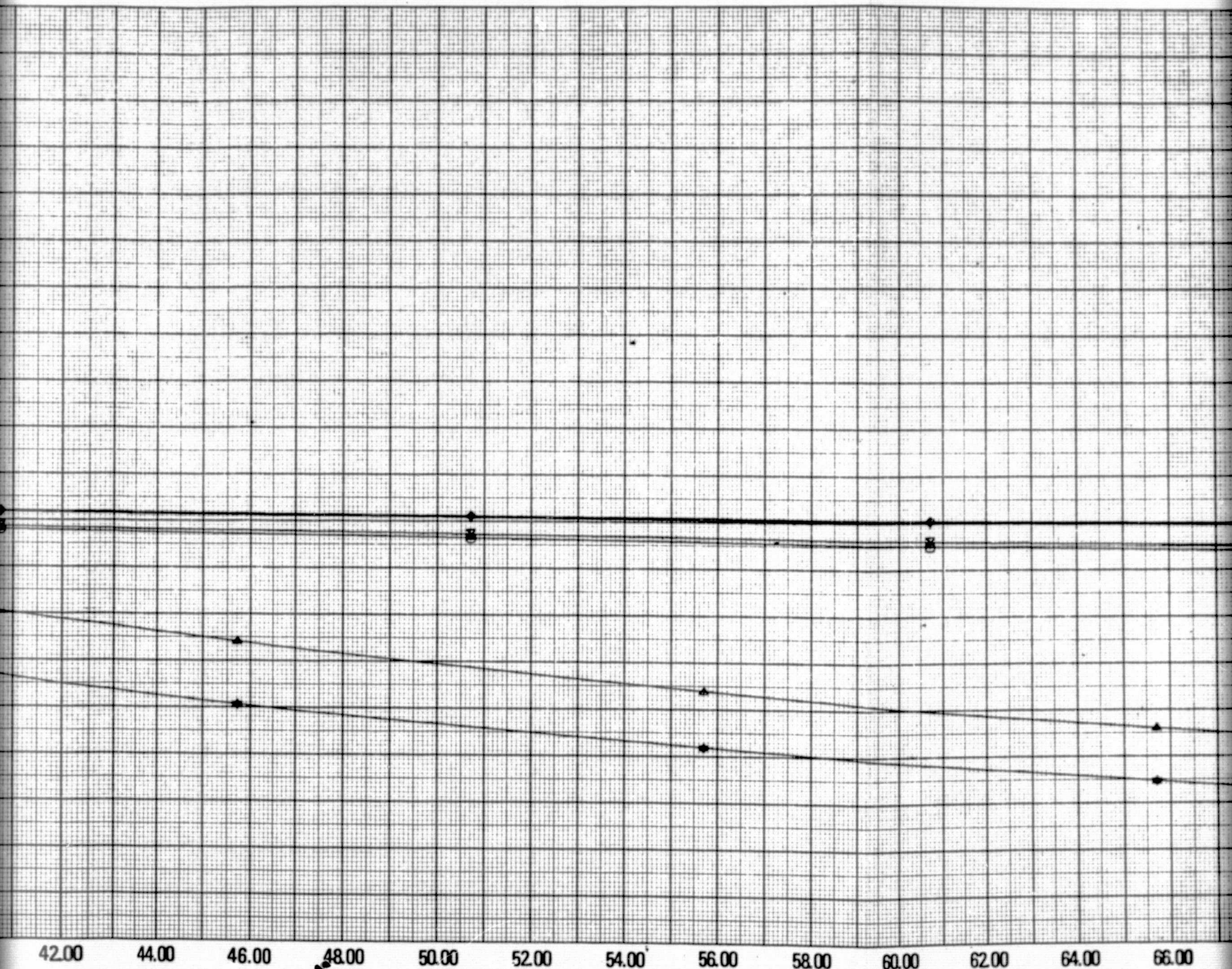

Figure 15. Voltage
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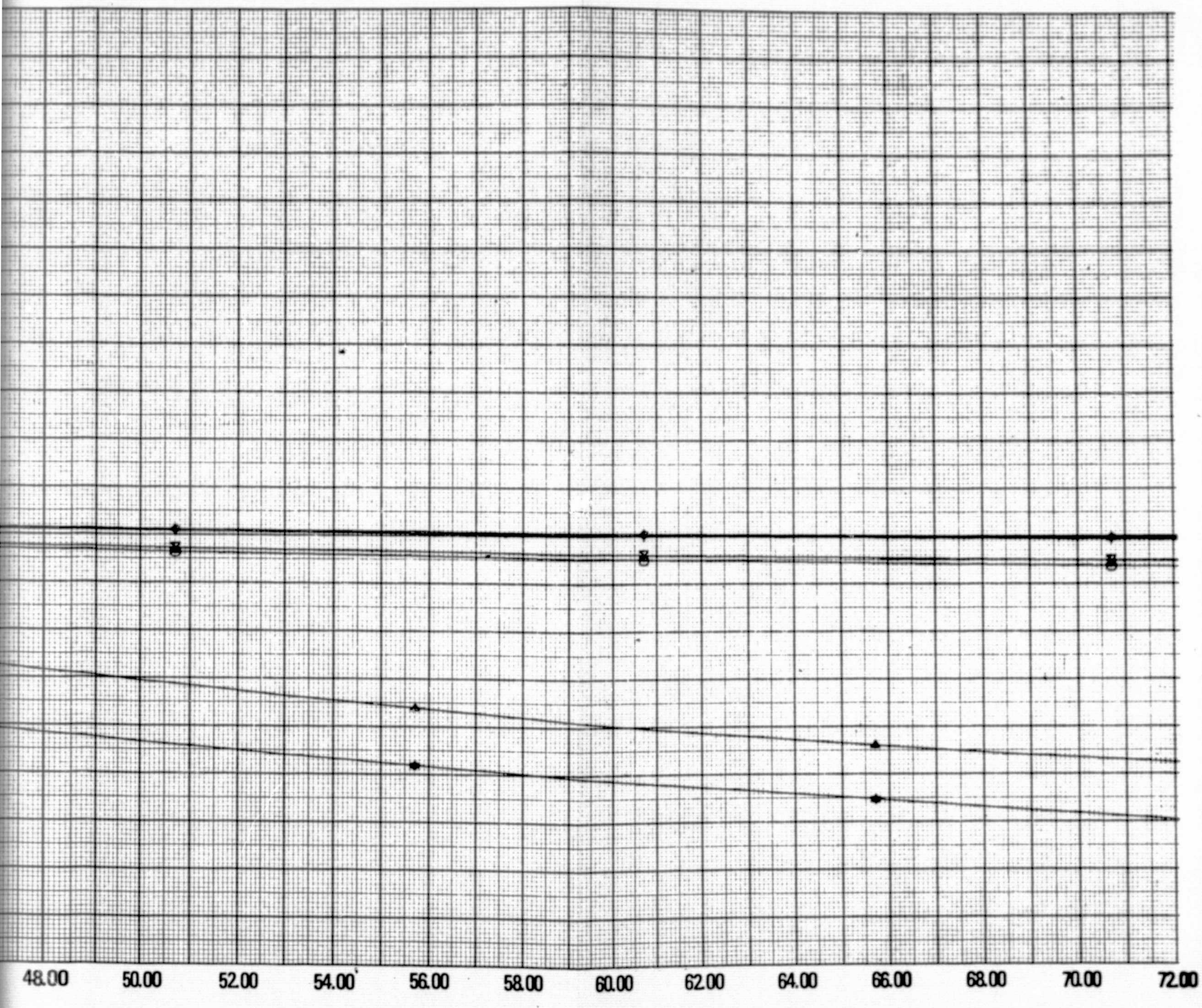

Figure 15. Voltage Decay Data, Test Sequence No. 1, Subgroup 2C

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It may be seen that the curves for cells with 1000 ohms resistors are only slightly below those for 10K ohm resistors, with the difference being about 12 mV at 72 hours (less at 24 and 48 hours). However, the spread between cells with identical resistors was only about 3 mV at 72 hours on this test, thus allowing a 12 mV difference to appear significant.

A semi-log plot for Group 2B (no resistors) and for those pairs of cells in Group 2C having 1000 and 100 ohms attached is shown in Figure 16. It would appear that whereas the 1000 ohm load may not be statistically distinguishable from no load, a 500 ohm load should be clearly detectable when adequate data is taken and plotted.

The second Voltage Decay Test done of Group 2 was performed without a charge-discharge cycle following the first test on this group. A semi-log plot of the data for Group 2A is shown in Figure 14. Note that the line is straighter and the slope is greater for the second test, with the voltages becoming equal after 72 hours. Thus, the prior 72 hour open circuit stand and 4 hour shorted period did not appear to degrade the performance on the second Voltage Decay Test appreciably. The response was about the same as that observed during an open circuit stand after inserting a single brief charge equal to the sum (in ampere hours) of the charges inserted in the two separate tests. These findings may prove useful for retesting of cells.

The second part of Test Sequence No. 1 (Test Procedure, Section 3) was done after the cells in Group 1 and 2 had been subjected to an Acceptance Test at TRW Systems, which in this case included three cycles for capacity determination, a 30 cycle "burn-in" sequence, and three more cycles for capacity redetermination. A Voltage Recovery Test was performed as an extension of the last cycle of the Acceptance Test series, as described in the procedure. Note also that a charge rate of C/2 was used for the charge injection on half of the cells after acceptance testing. This was done to obtain a comparison with results using a C/10 charge rate under controlled conditions.

A representative set of voltage vs. time curves (for Group 1B) for the latter portion of the Voltage Recovery Test (Procedure, Para. 3.1) showing the response above 1 volt, is shown in Figure 17. In this test,

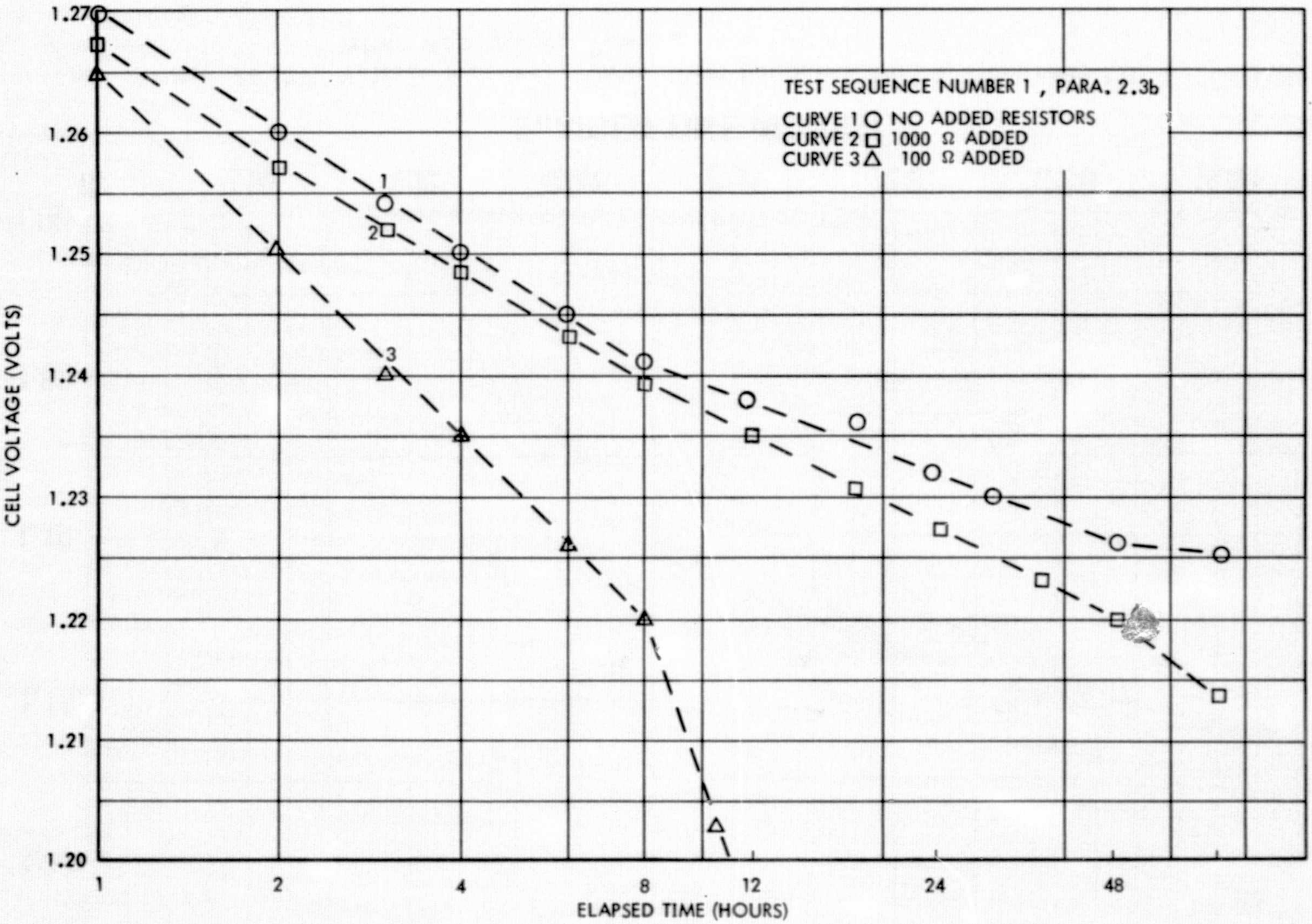

Figure 16. Semi-log Plot of Data from Figure 15

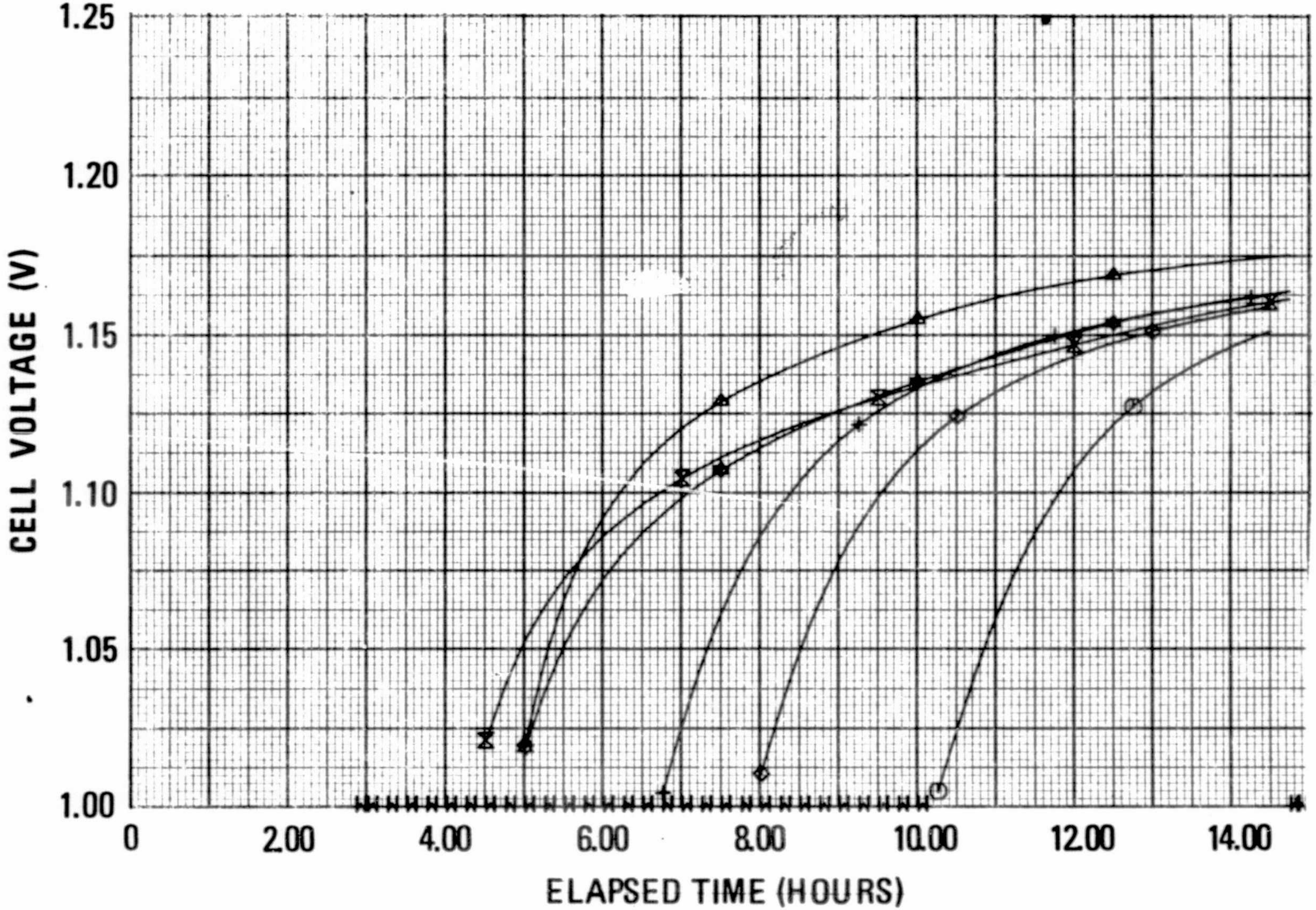

Figure 17. Voltage Recovery Data, Test Sequence No. 1, Subgroup 1B, After Acceptance Testing

the open circuit period was not a full 24 hours, as the open circuit period was terminated at 12 hours when the last cell voltage reached 1.15 volts. Note the large spread in time to reach 1 volt (6 hours). More complete data for this type of test was obtained during the other test sequences described below.

Figure 18 shows the open circuit voltages of the six cells in Group 1B following a 6 minute charge at the C/10 rate. These voltages after acceptance testing were 10-20 mV lower than those from the same test prior to acceptance testing (e.g., Figure 13).

The cell voltage curves for cells in Group 1A with certain resistors attached is shown in Figure 19. In these tests the resistances added were changed from those used in earlier tests (100, 1000, and 10K ohms) to 100, 330, and 1000 ohms to provide more data in what appeared to be a more useful range.

Comparison of Figure 19 with Figure 18 shows that, as before acceptance testing, the difference between the curves for 1000 ohms and for no added resistor is very small. The effects with 330 ohms are much more apparent. It is interesting to note than in spite of the obvious divergence of the curve for 330 ohms in Figure 19, the voltages for this resistance were at or above 1.17 volts at the end of 24 hours, and hence cells with this order of internal shorting resistance would pass the test if a 1.17 volt end-point criterion were used.

The same type of information for use of a 12 minute charge at the C/10 rate for Voltage Decay testing after acceptance testing of Group 2 cells is shown in Figure 20 and 21. A semi-log plot of data from these runs showing the effect of various added resistors is shown in Figure 22.

These curves show that voltages after the longer charge were 20 mV greater at the beginning, and 10 mV greater after 48 hours, and that the voltages on 100 and 330 ohm resistors remained higher longer than after the 6 minute charge. The voltage from one cell with 330 ohms did not drop significantly below that for one cell with 1000 ohms throughout the stand time. Thus the test appears to be less sensitive to shorting loads after a 2 percent charge (12 min. at C/10) than after a 1 percent charge (6 min. at C/10).

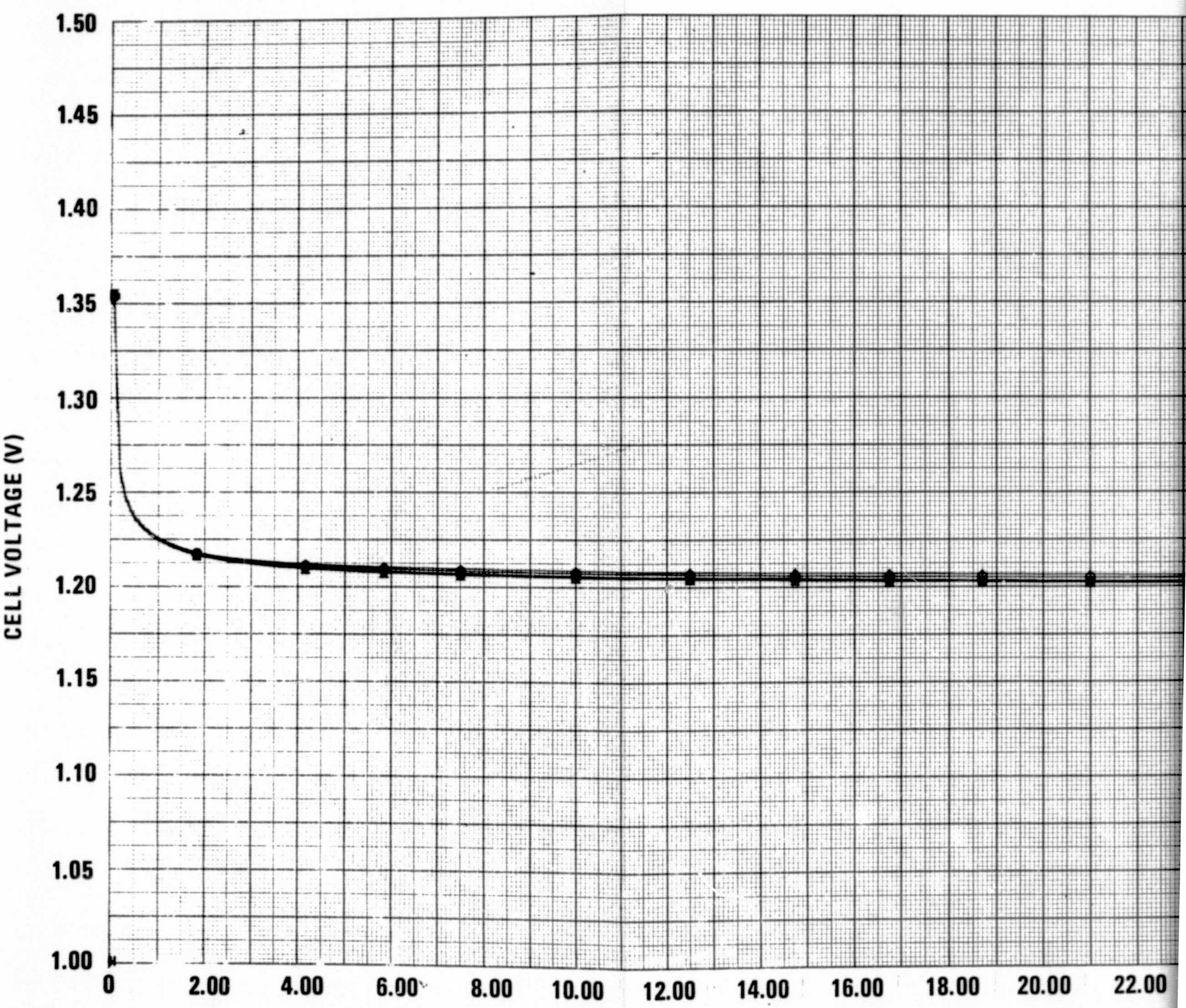

Figure 18. Voltage Decay Data, Test Sequence No. 1, Subgroup 1 B, After Acceptance Testing

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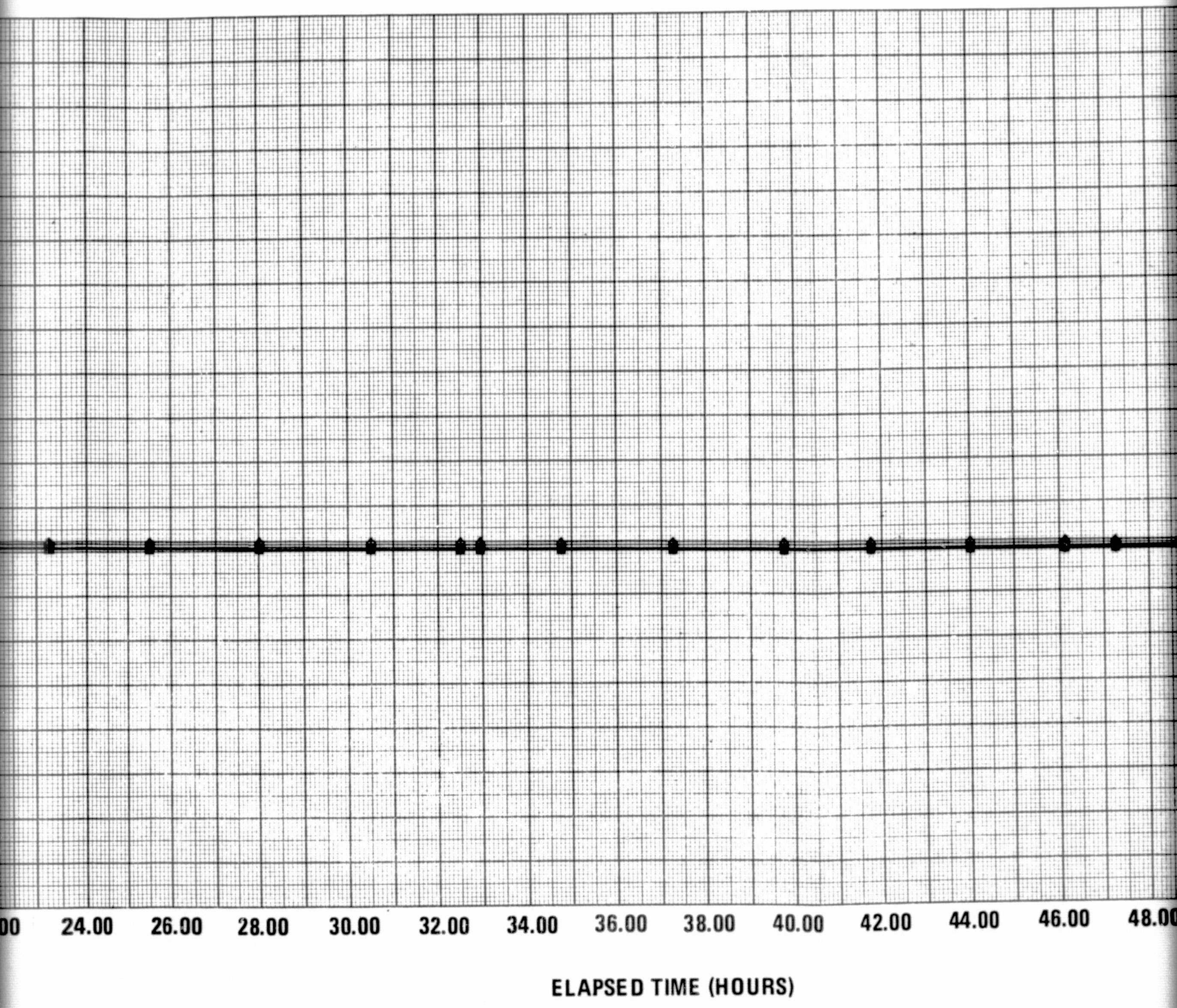

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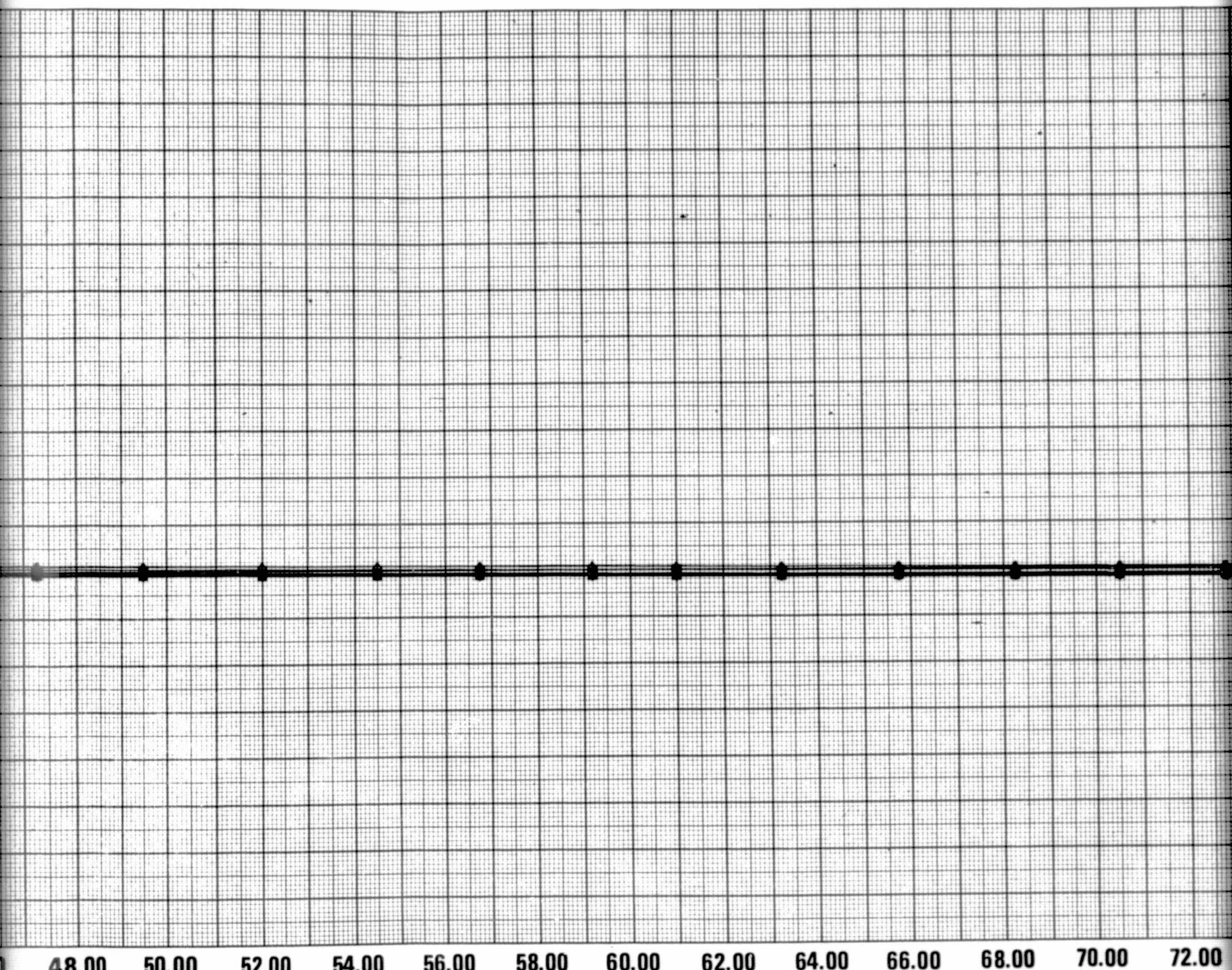

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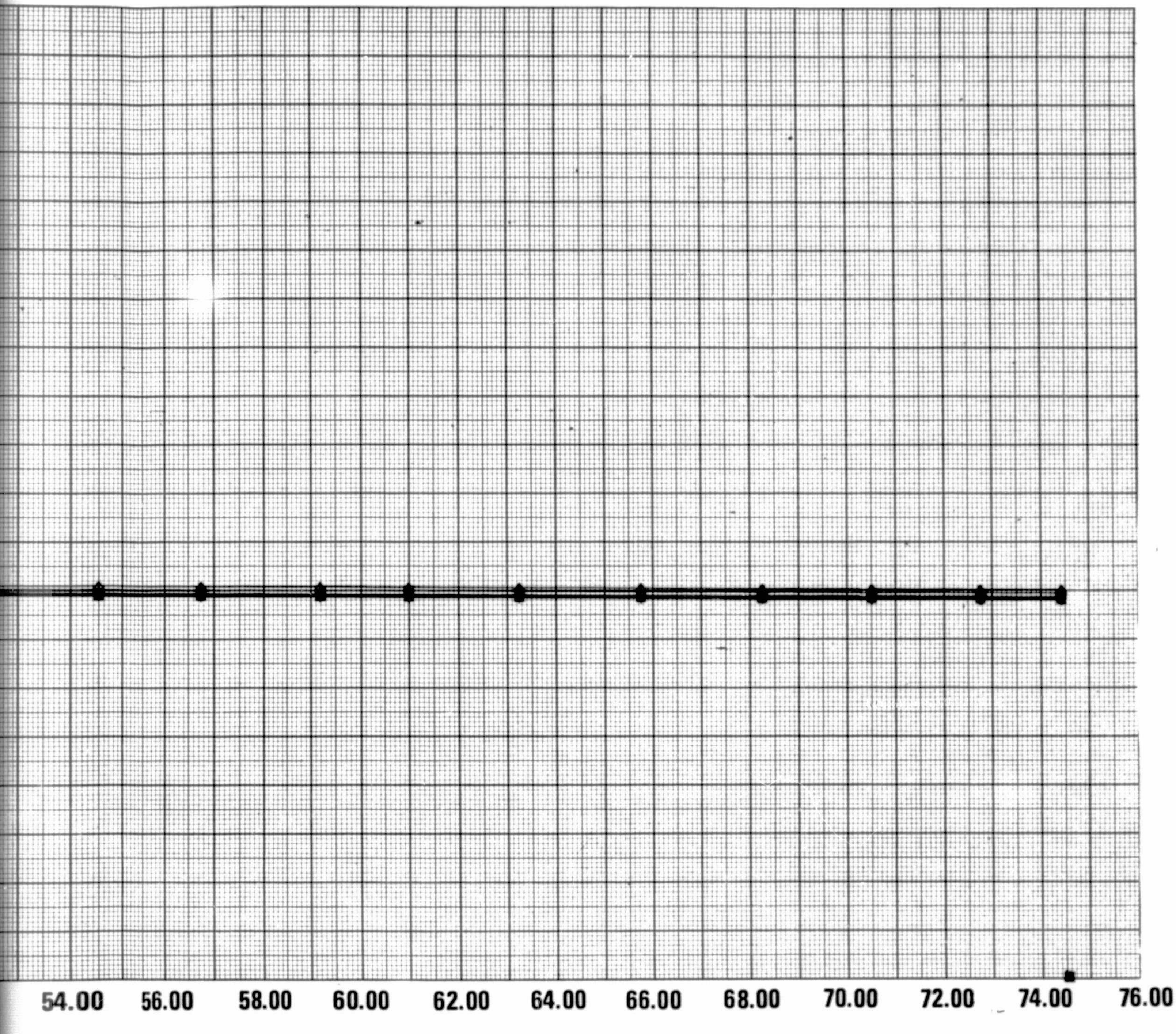
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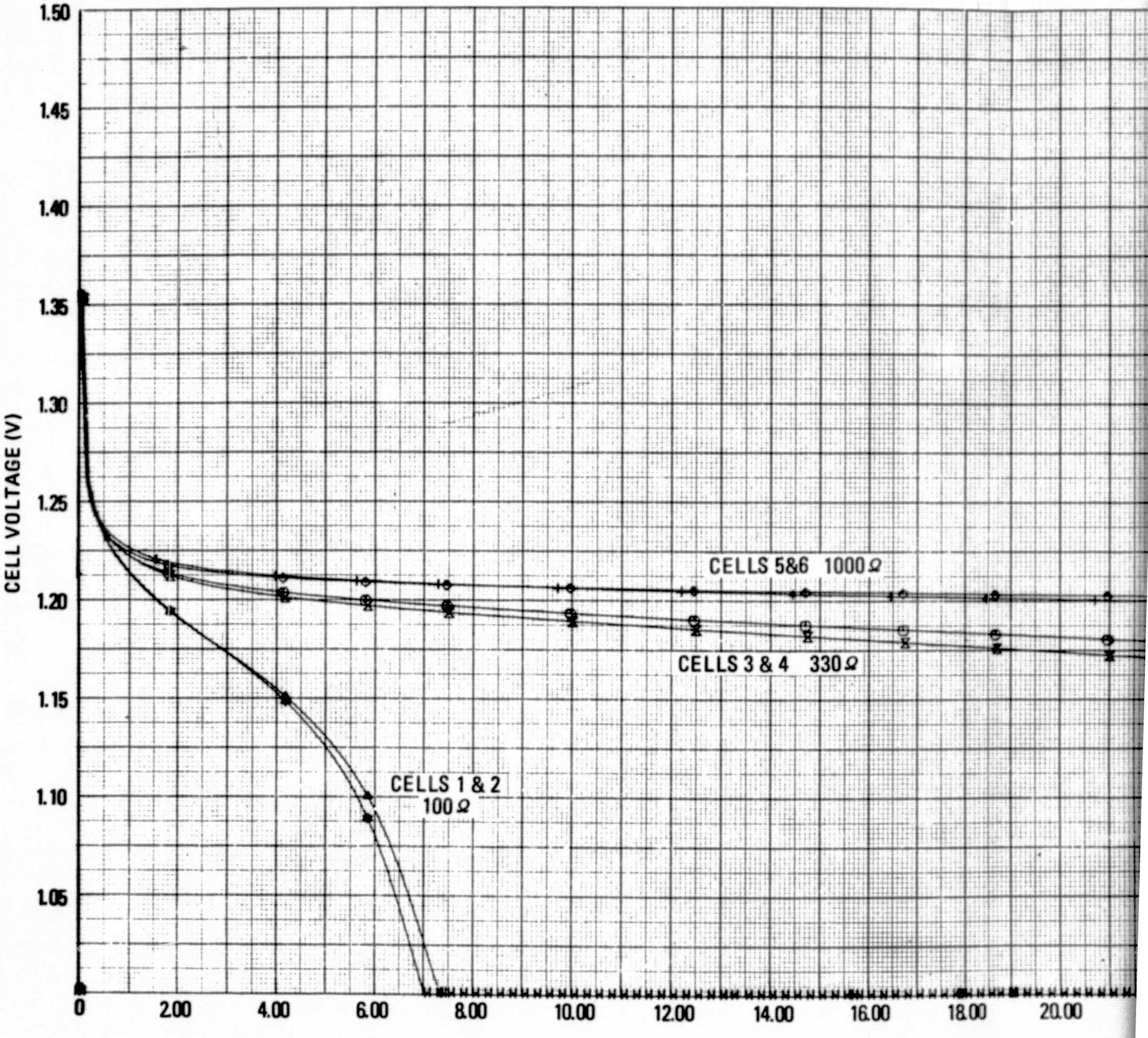

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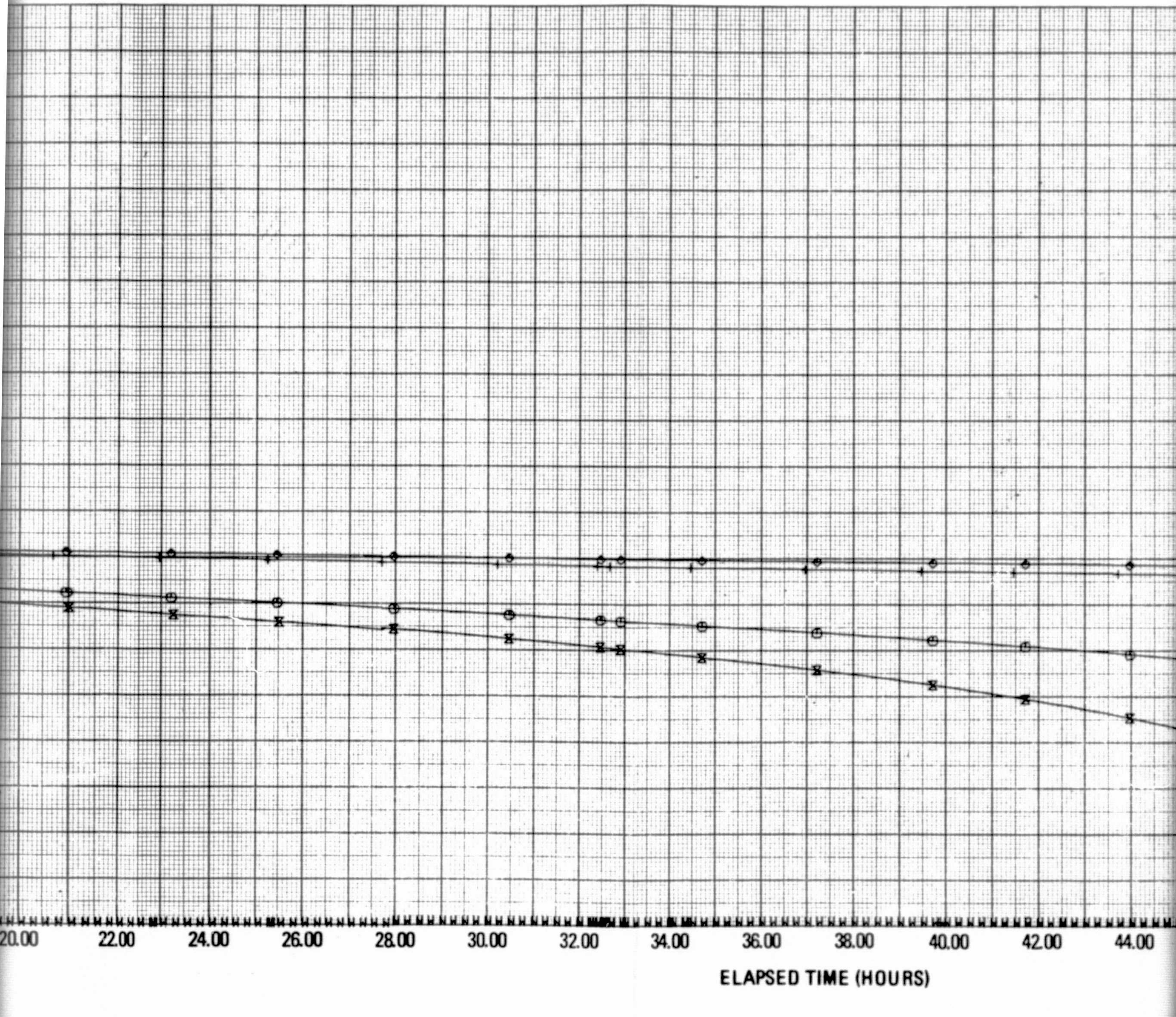

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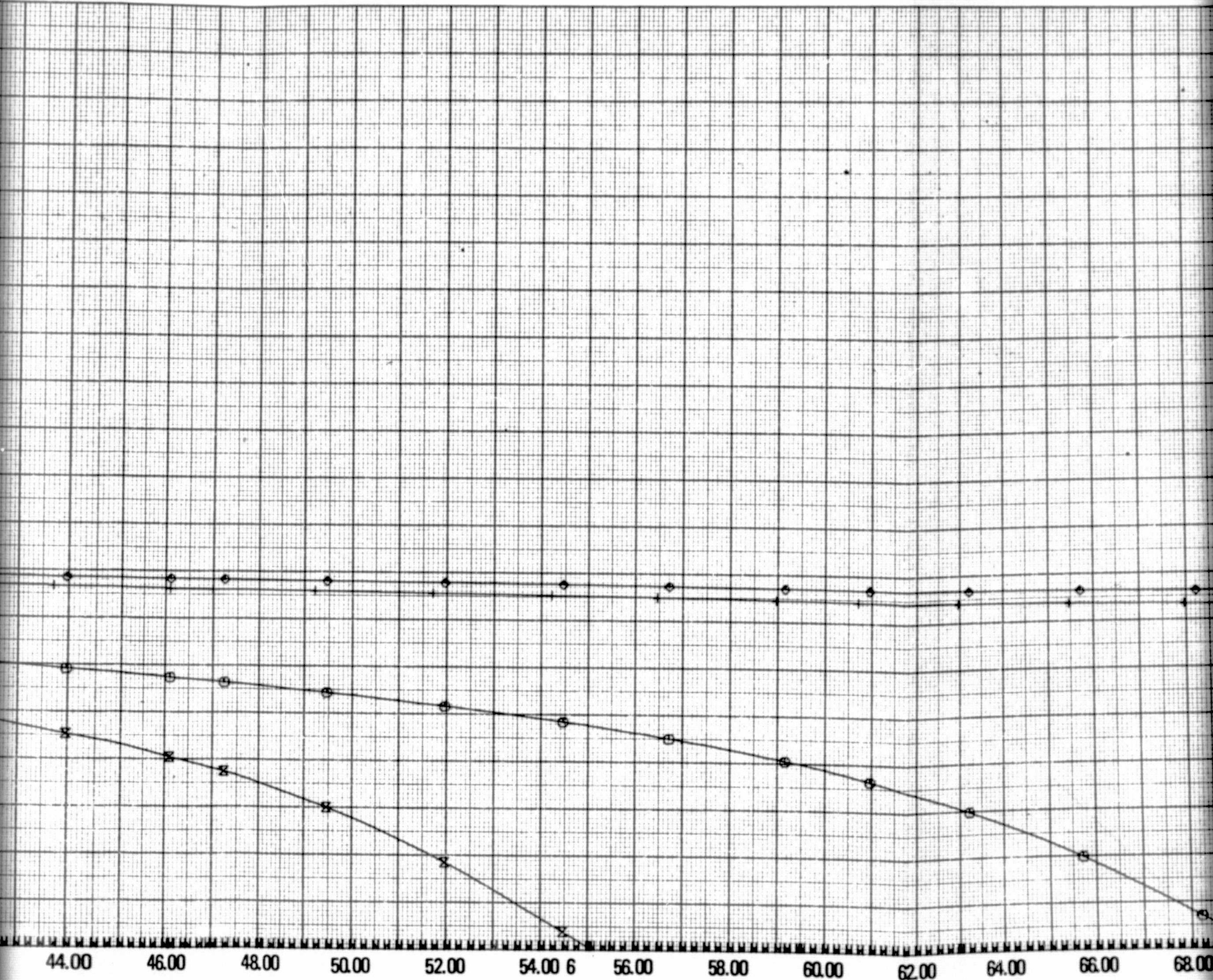

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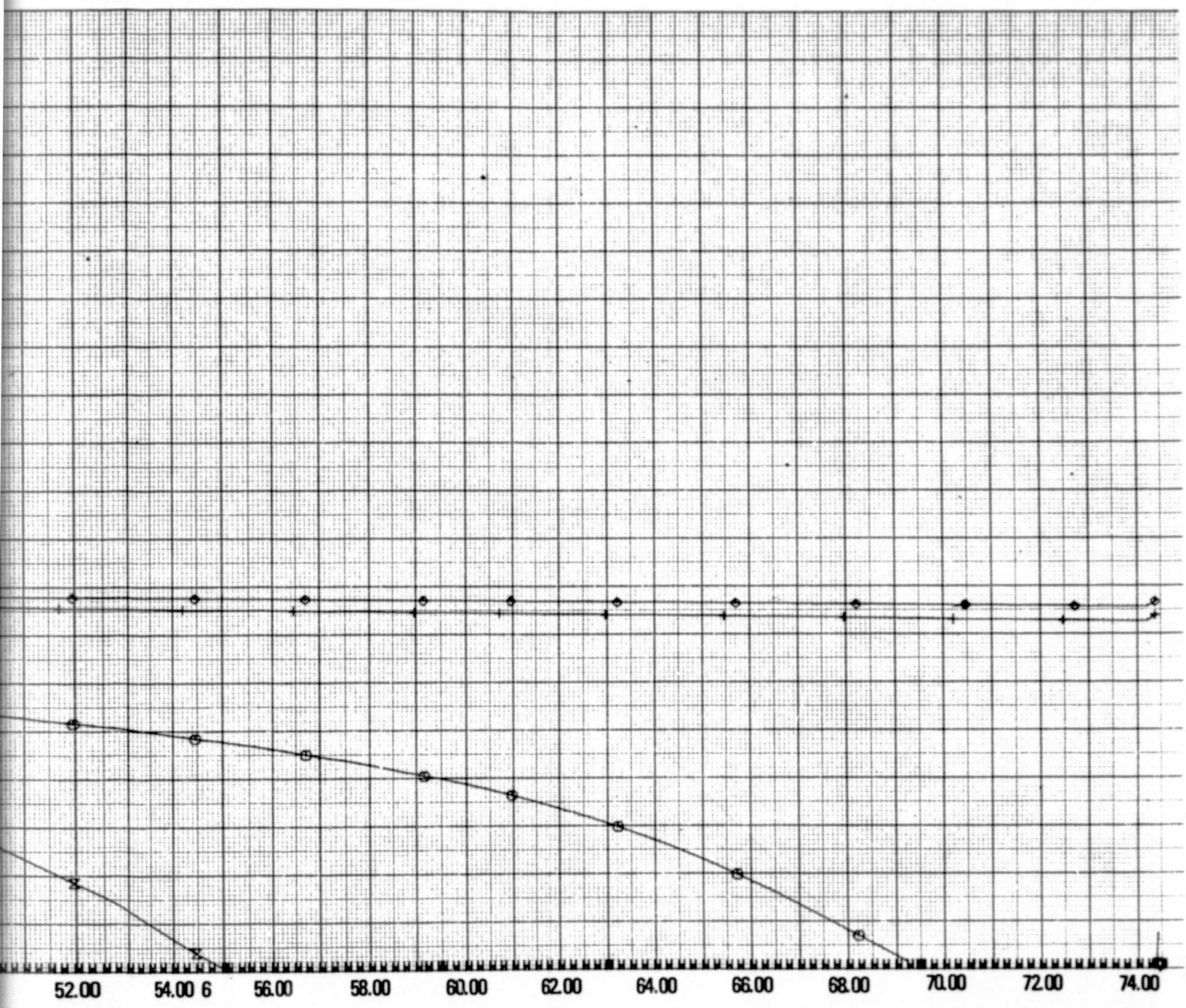
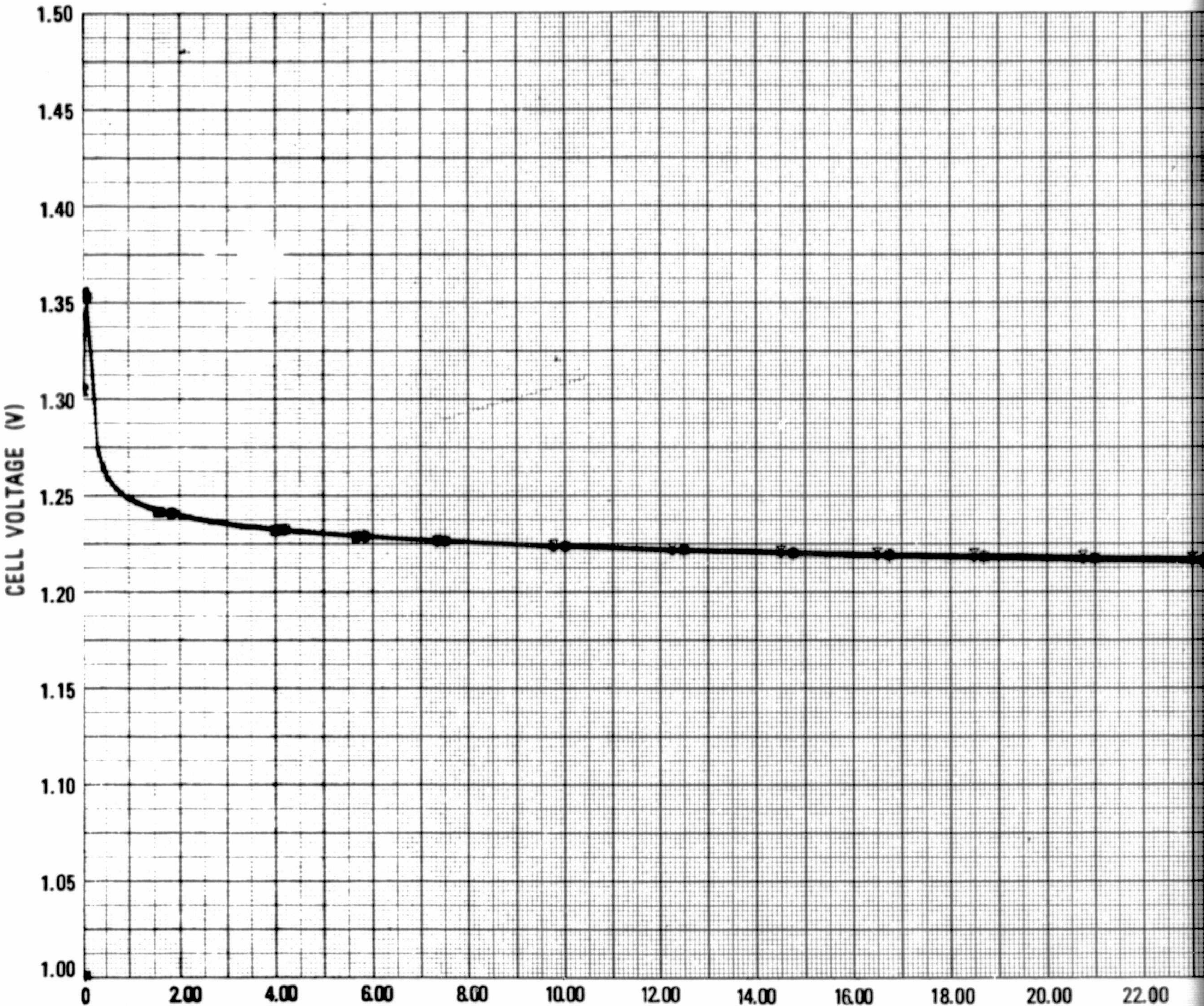


Figure 19. Voltage Decay Data, Test Sequence No. 1, Subgroup 1A After Acceptance Testing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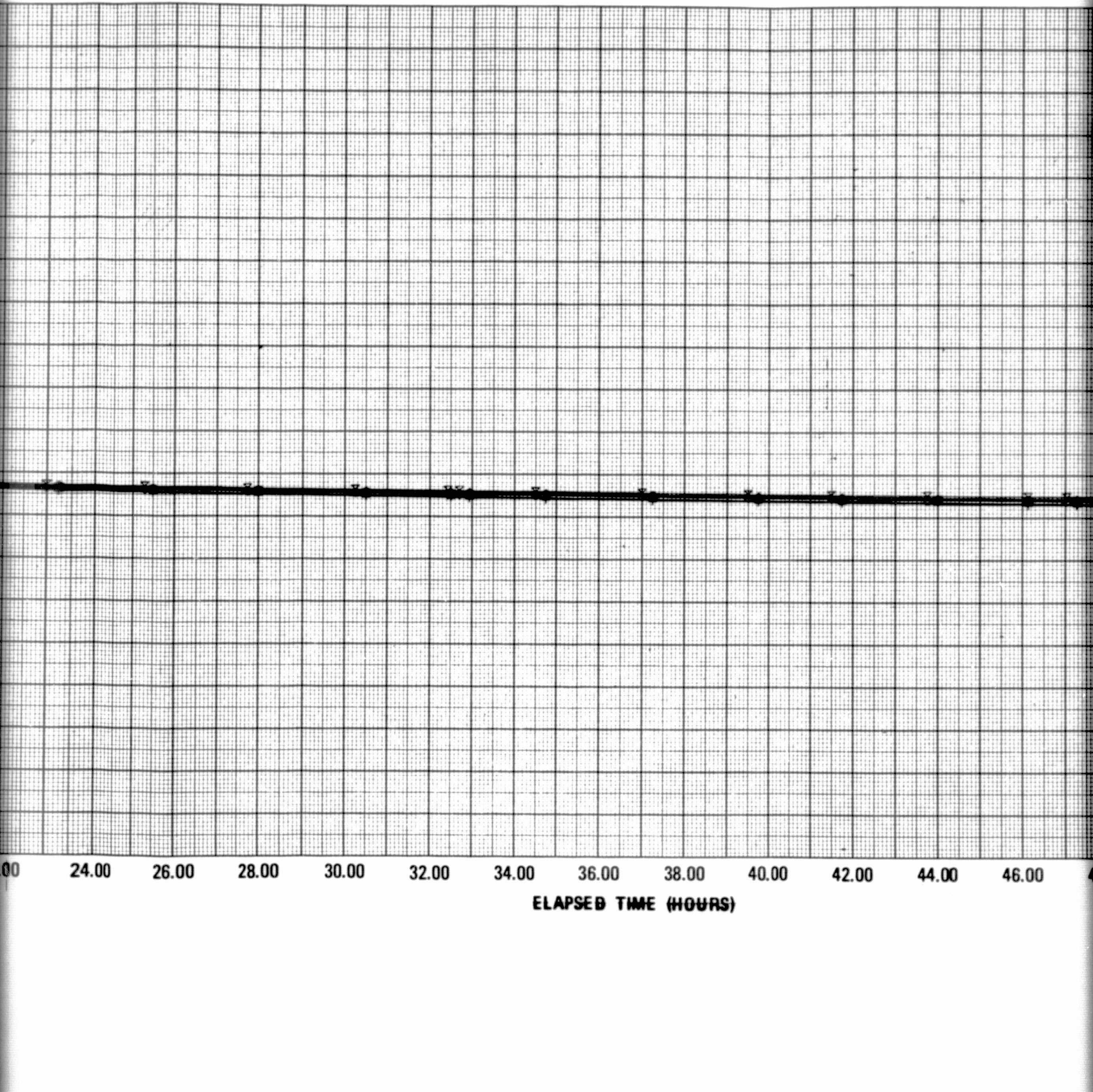

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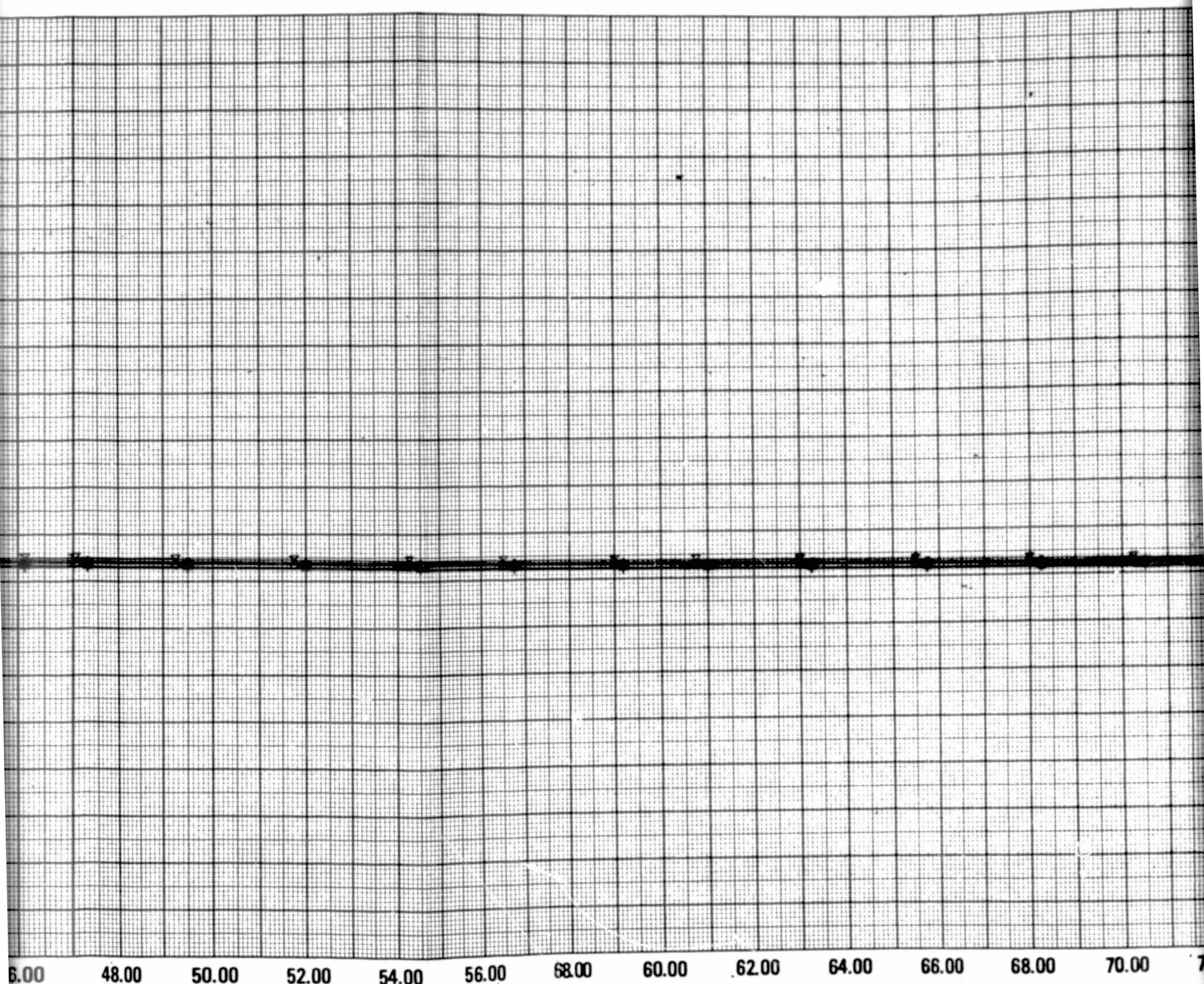

Figure 20. Voltage Decay Data, Test Sequence No. 1, Subgroup 1D, After Acceptance Testing

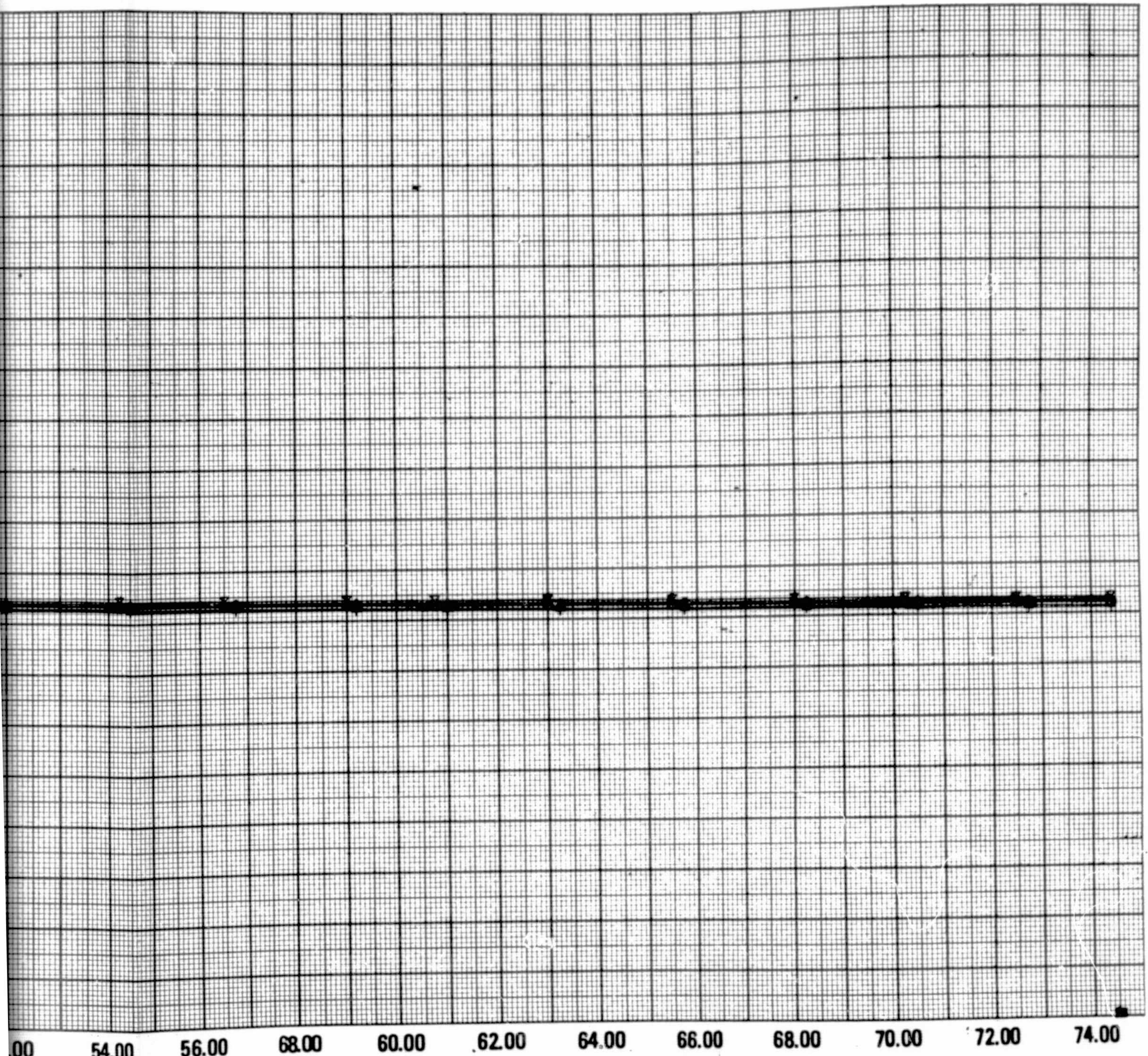

Figure 20. Voltage Decay Data, Test Sequence No. 1, Subgroup 1D, After Acceptance Testing

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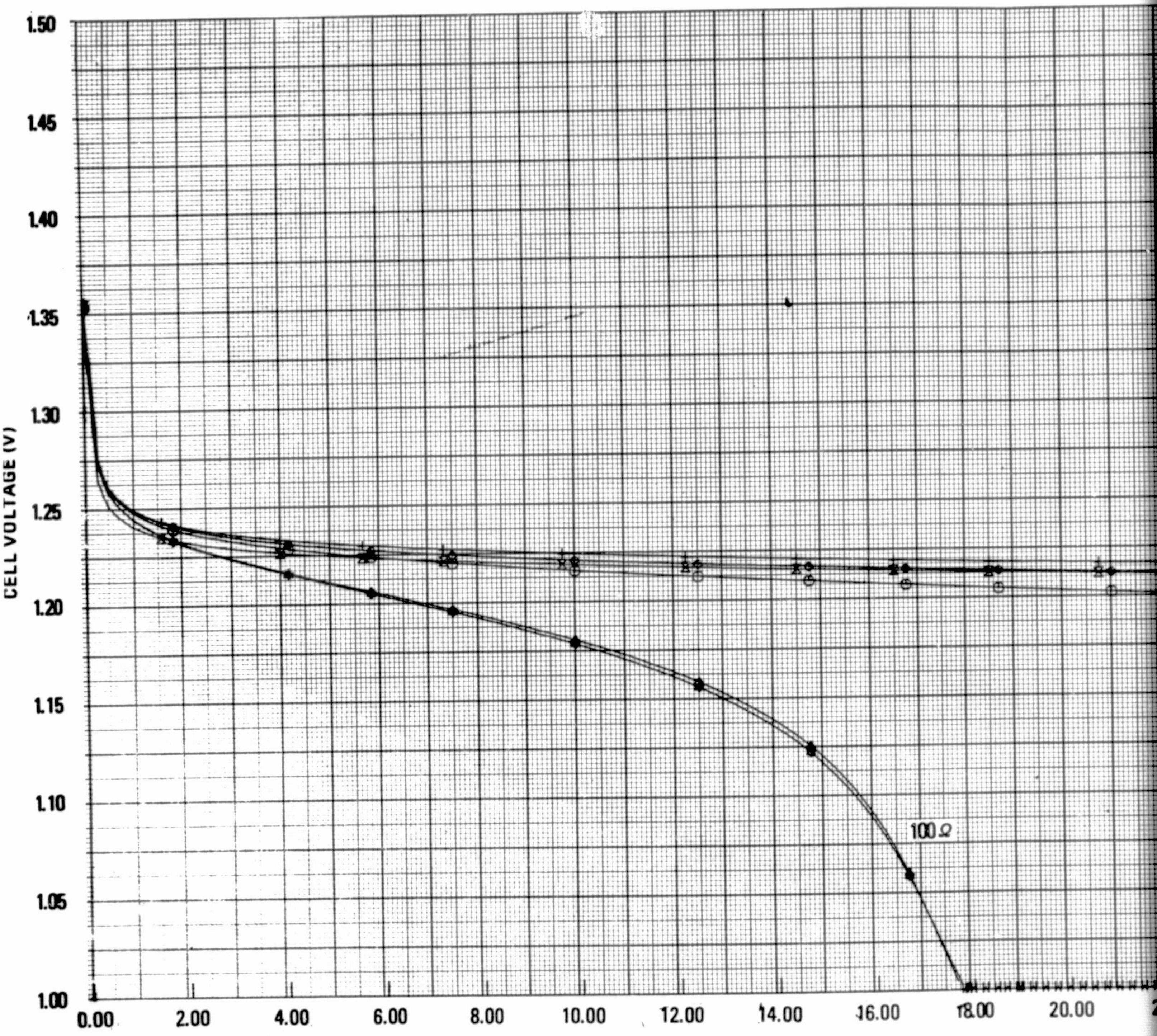

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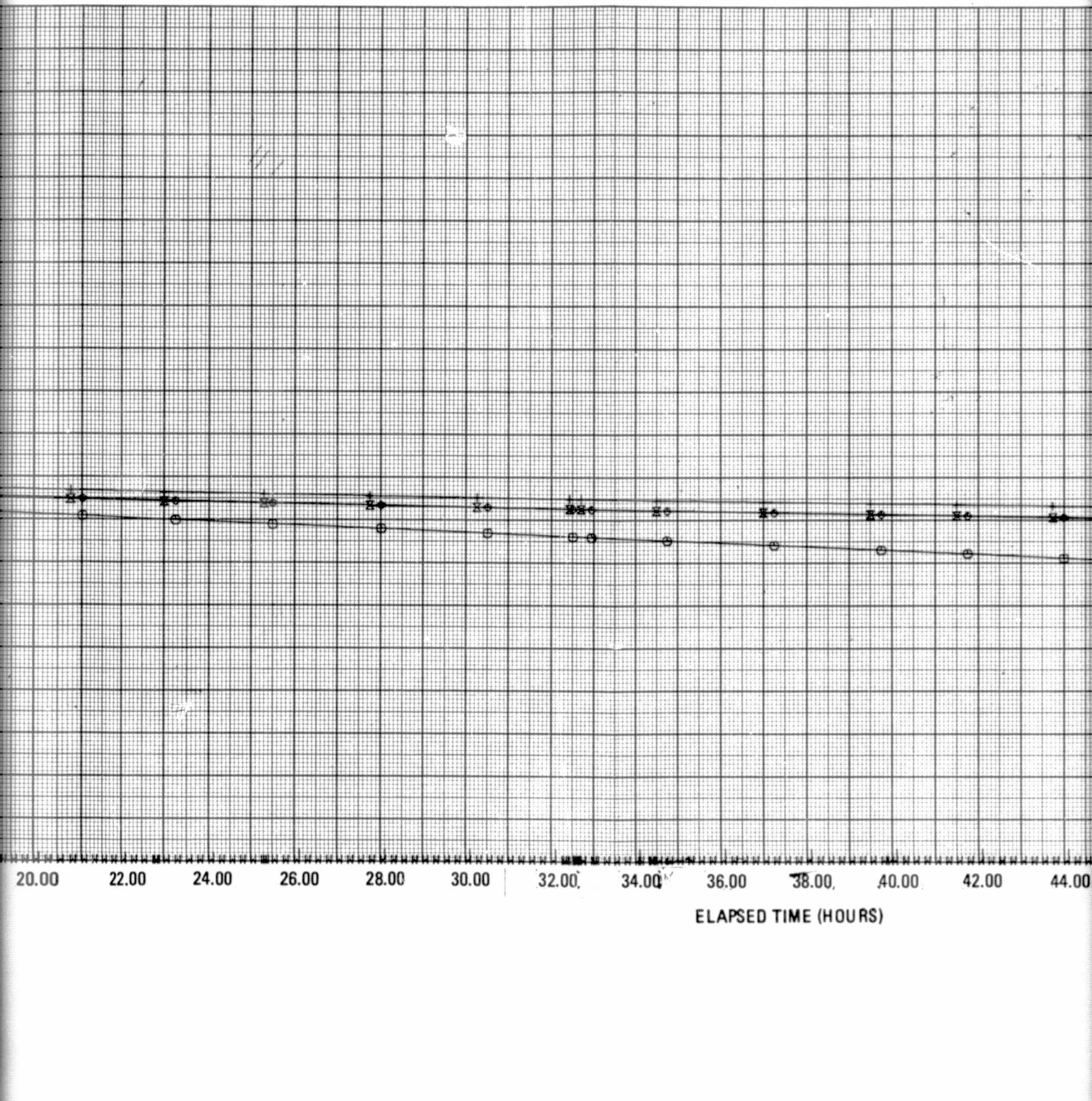

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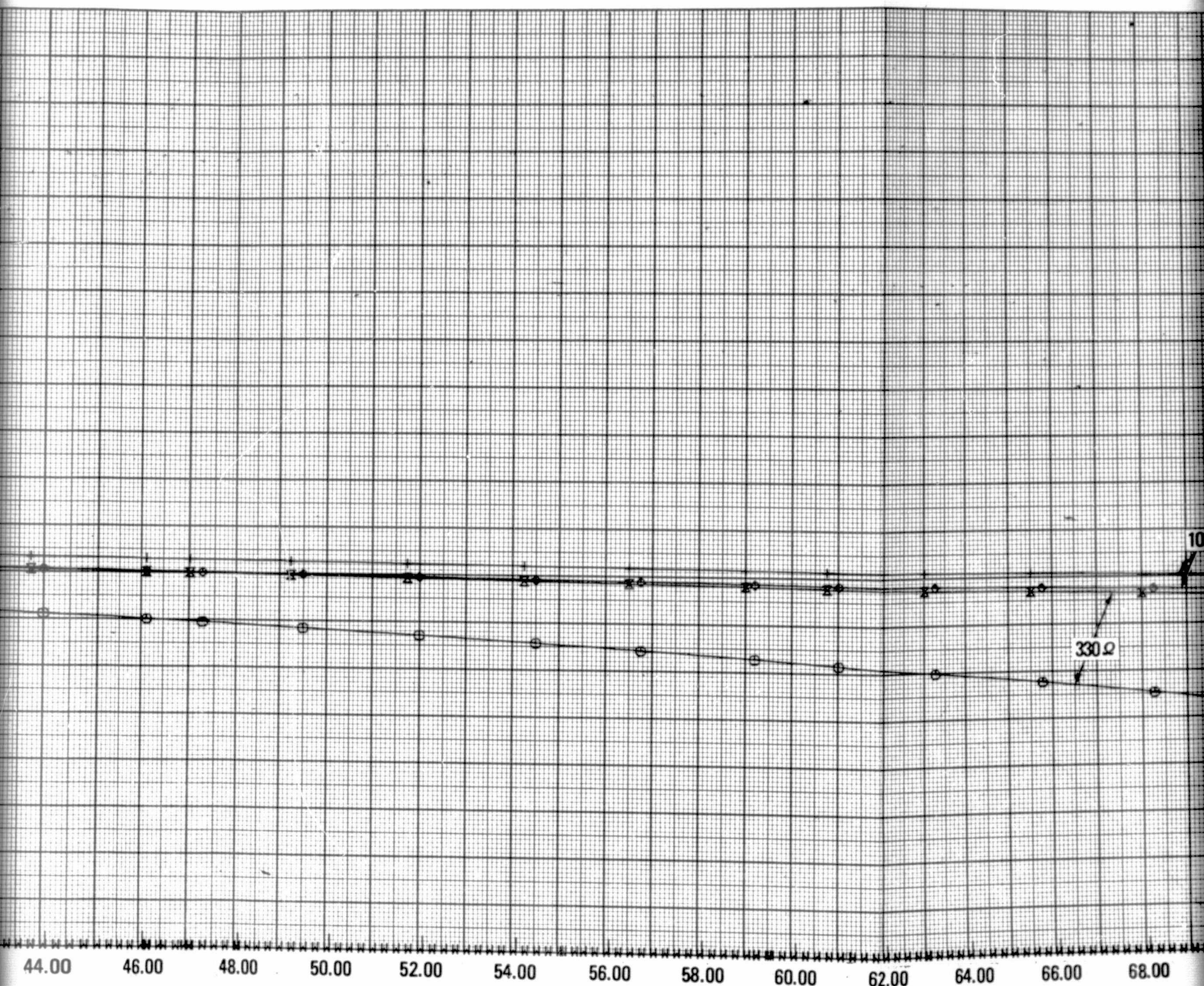

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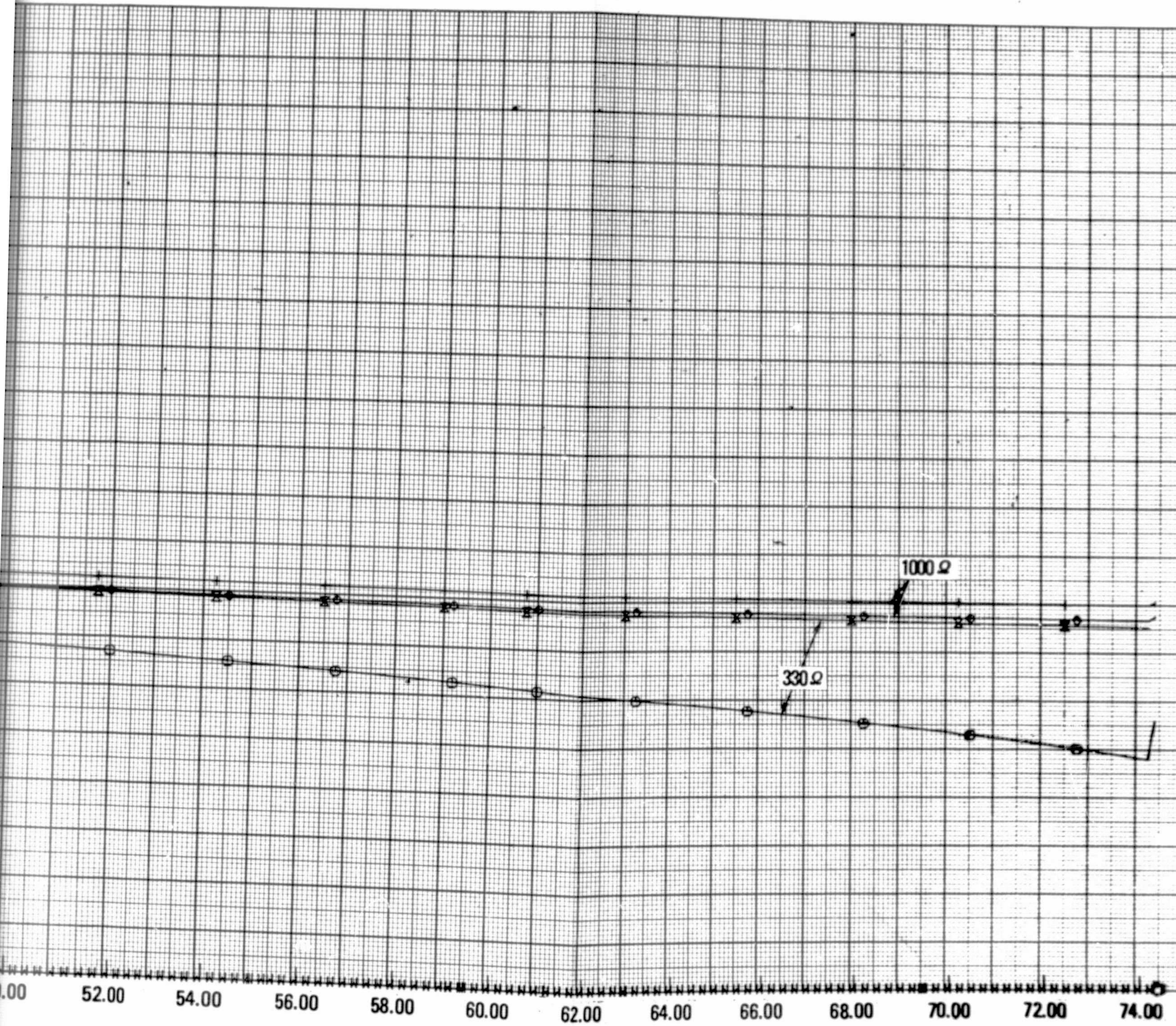

Figure 21. Voltage Decay Data, Test Sequence No. 1, Subgroup 1C, After Acceptance Testing

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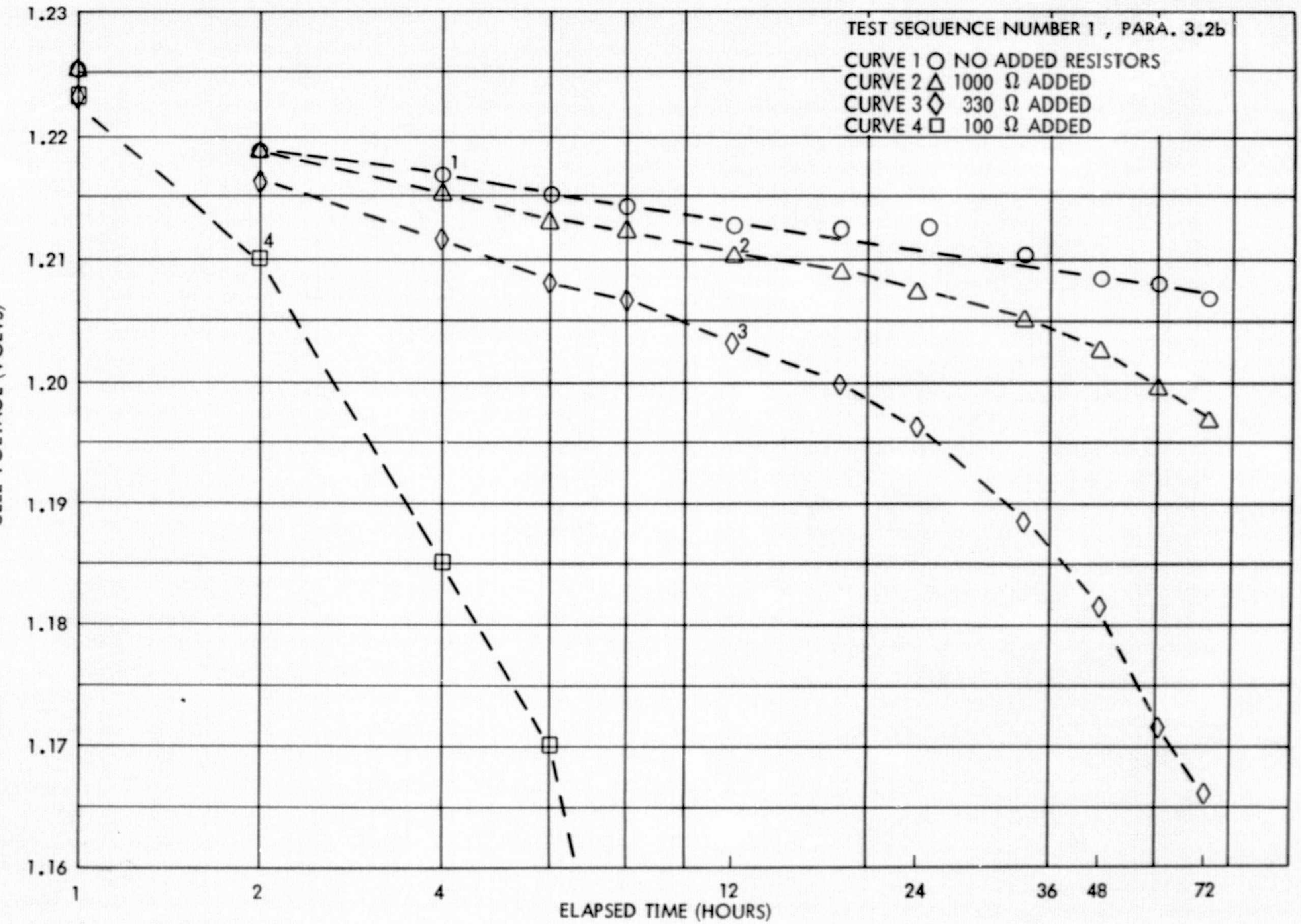

Figure 22. Semi-log Plot of Data From Figure 21

Linear time base data plots for the four groups tested using a C/2 charge data and the same ampere hour throughout as above are shown in Figures 23 through 26. It may be seen that the curves for no external resistor and for 1000 ohms added are identical within a few millivolts to the corresponding curves for a C/10 charge rate (i.e., in Figures 18 through 21). Comparison of Figures 24 and 19 shows that after the C/2 charge the test was not as sensitive to the resistors as after the C/10 charge, whereas the scatter of the data was about the same for both charge rates.

4.3.2 Results From Test Sequence No. 2

Test Sequence No. 2 was performed on some Gulton 12 Ah cells that had been in continuous shorted storage for about 1 year. These cells were tested to see if any difference could be seen between the way Gulton and General Electric cells respond to short testing, and to begin to gather data on the effects of prolonged storage. These cells were the newest Gulton cells available for test.

The first test performed on this test lot was a "voltage recovery" test done by merely removing the shorts and following the voltages, i.e., without prior cycling. At the end of 24 hours on open circuit the voltages ranged from 0.03 to 0.15 volt. This response is similar to that obtained from the cells at the beginning of Test Sequence No. 1 after several weeks on continuous short using the same procedure. This data was not plotted.

Following a 4 hour short down, Voltage Decay testing was performed, without a prior charge-discharge cycle, using a 6 and a 12 minute C/10 charge on two different groups of 6 cells each. The voltage decay curves for the cells given a 12 minute charge are shown in Figure 27. Note the relatively rapid drop of all cells, to around 1.05 volts, in 24 hours. The voltages after the 6 minute charge decayed even more rapidly.

A plot of highest and lowest voltages from Figure 27, versus log time, is shown in Figure 28. The dashed line shows the position of the response expected for non-shorted cells. Note that the slopes and shapes of the curves are similar to those for the 24 Ah cells with 100 ohms attached, shown in Figure 16. However, as shown below, no internal shorting was detected in these cells when the test was repeated after a conditioning cycle.

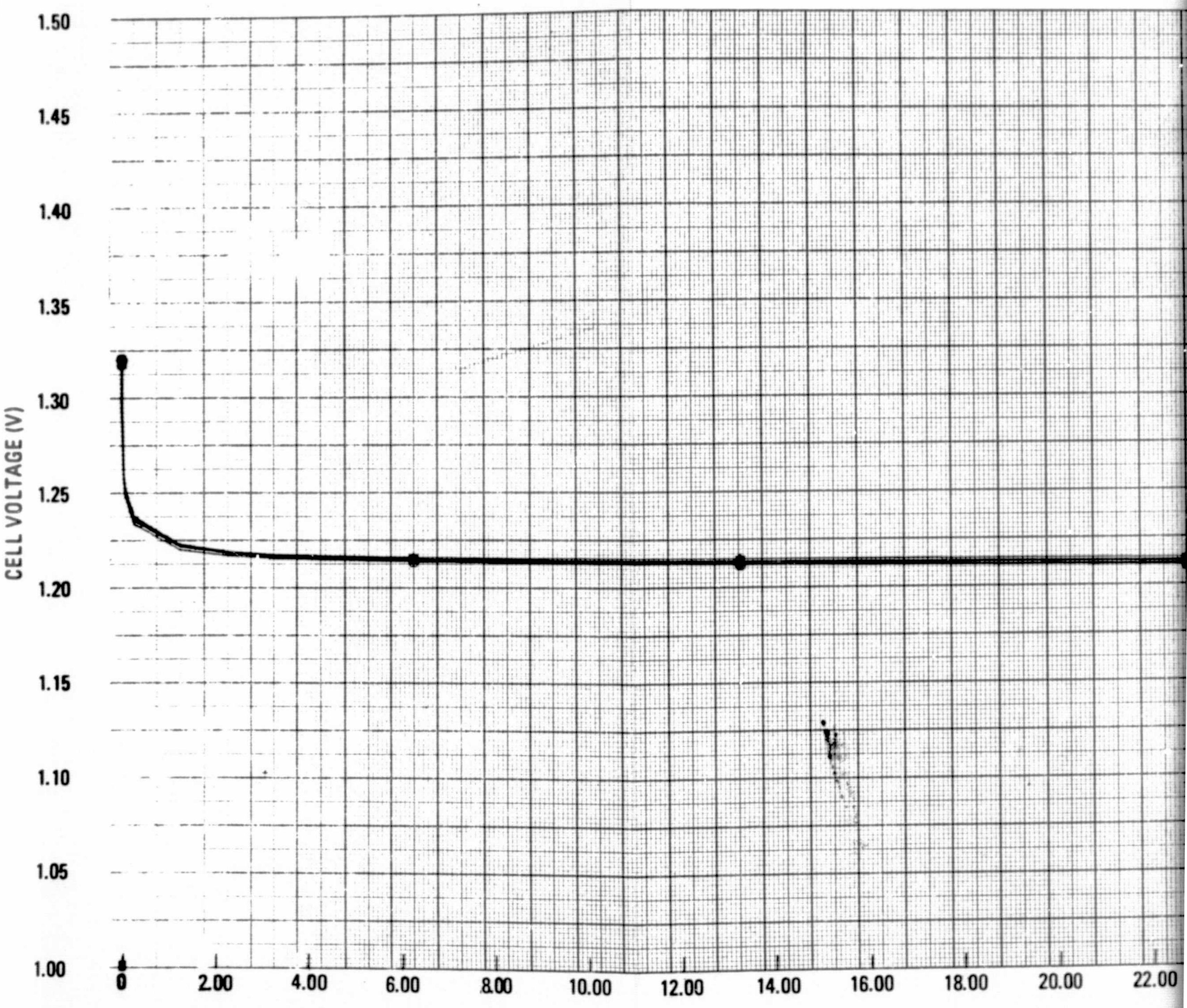

Figure 23. Voltage Decay Data, Test Sequence No. 1, Subgroup 2B After Acceptance Testing

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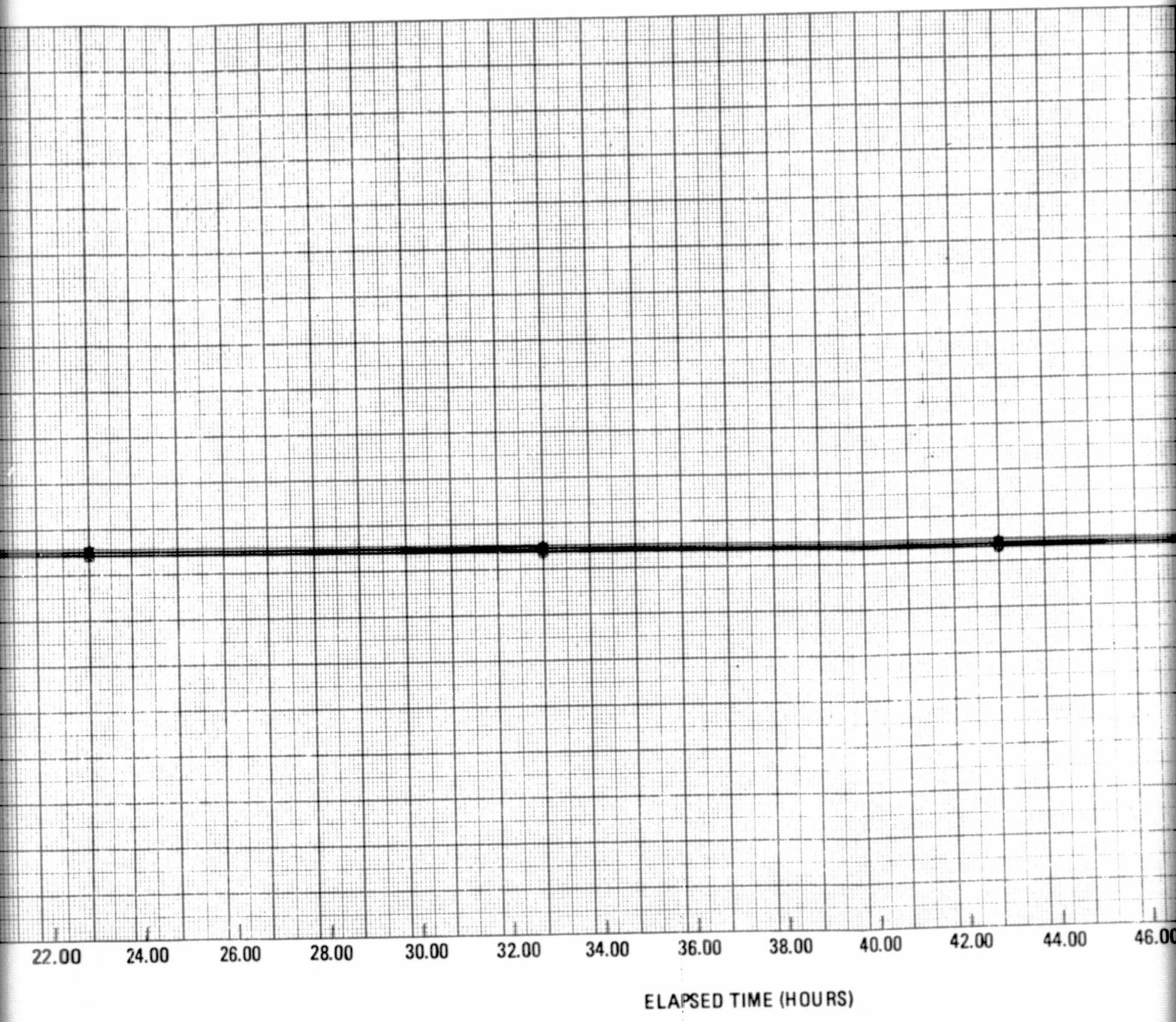

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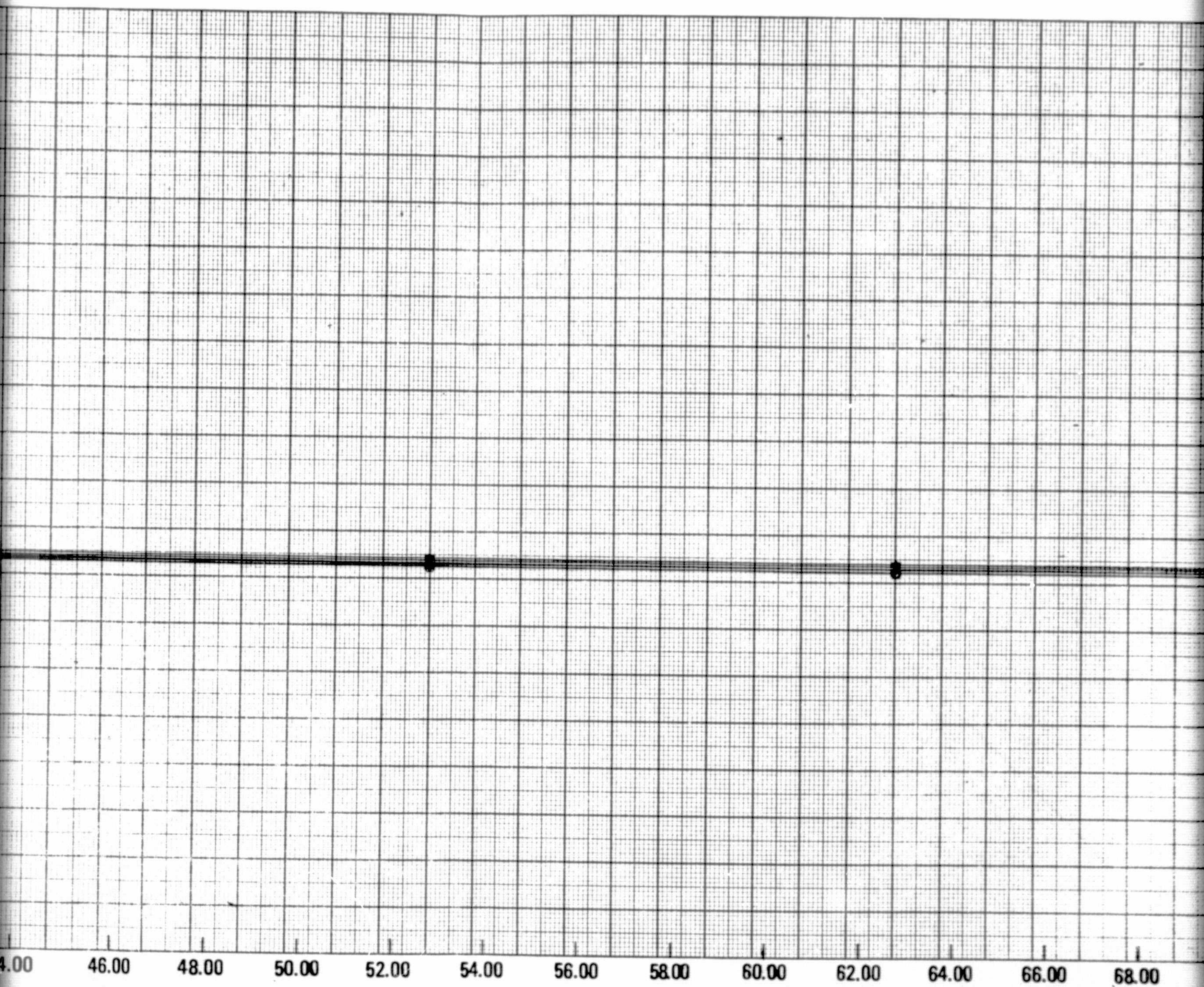

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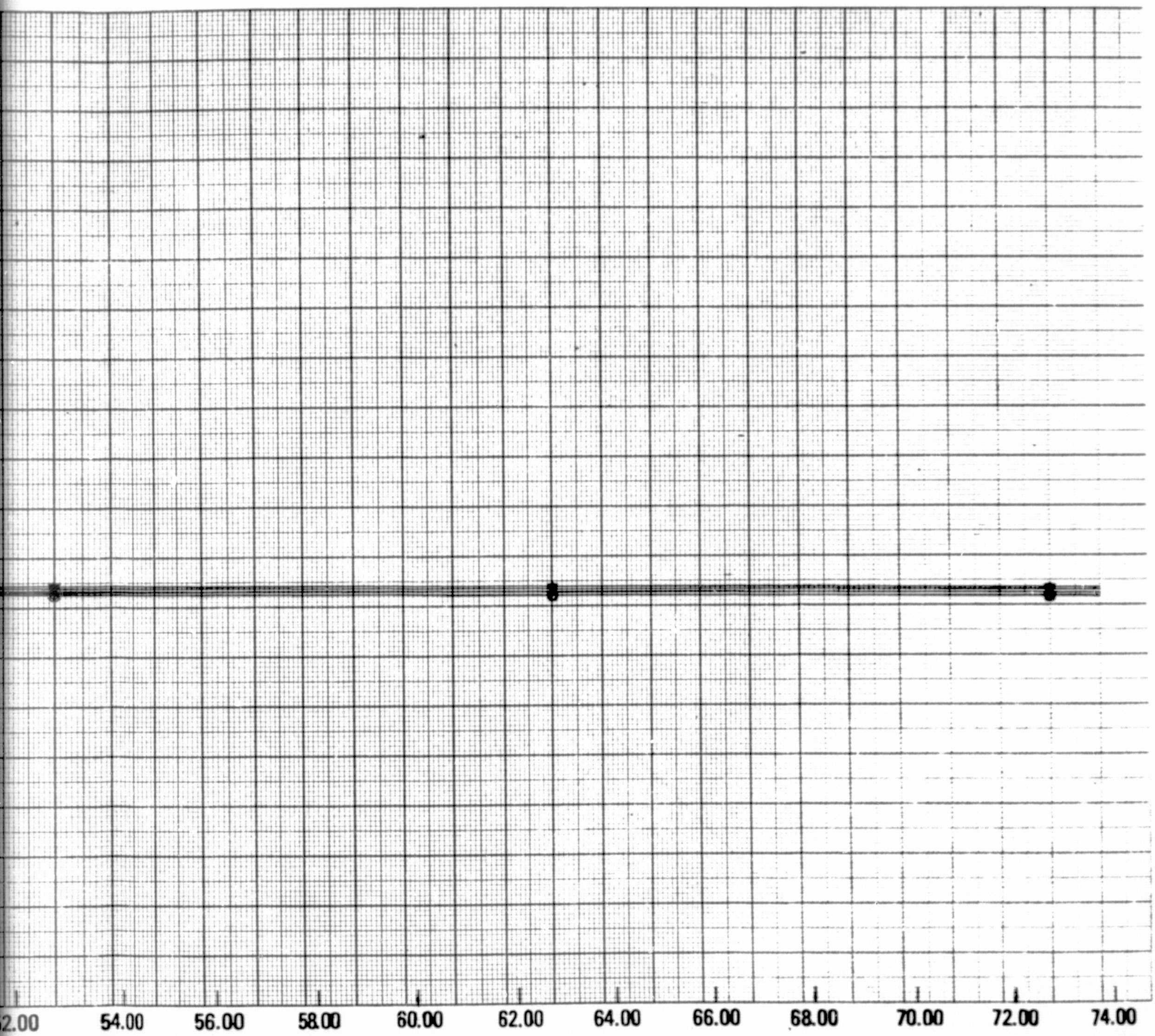

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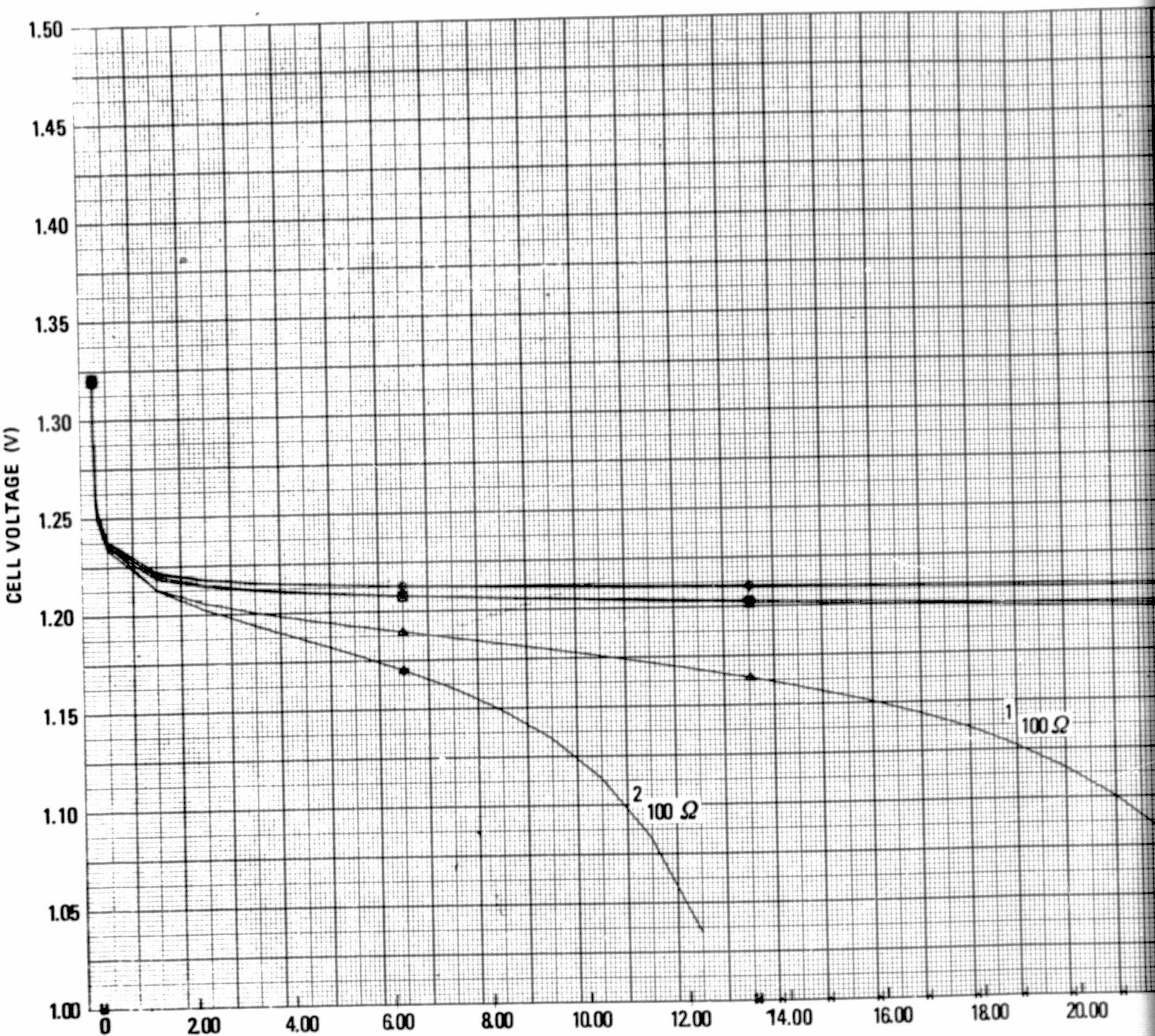

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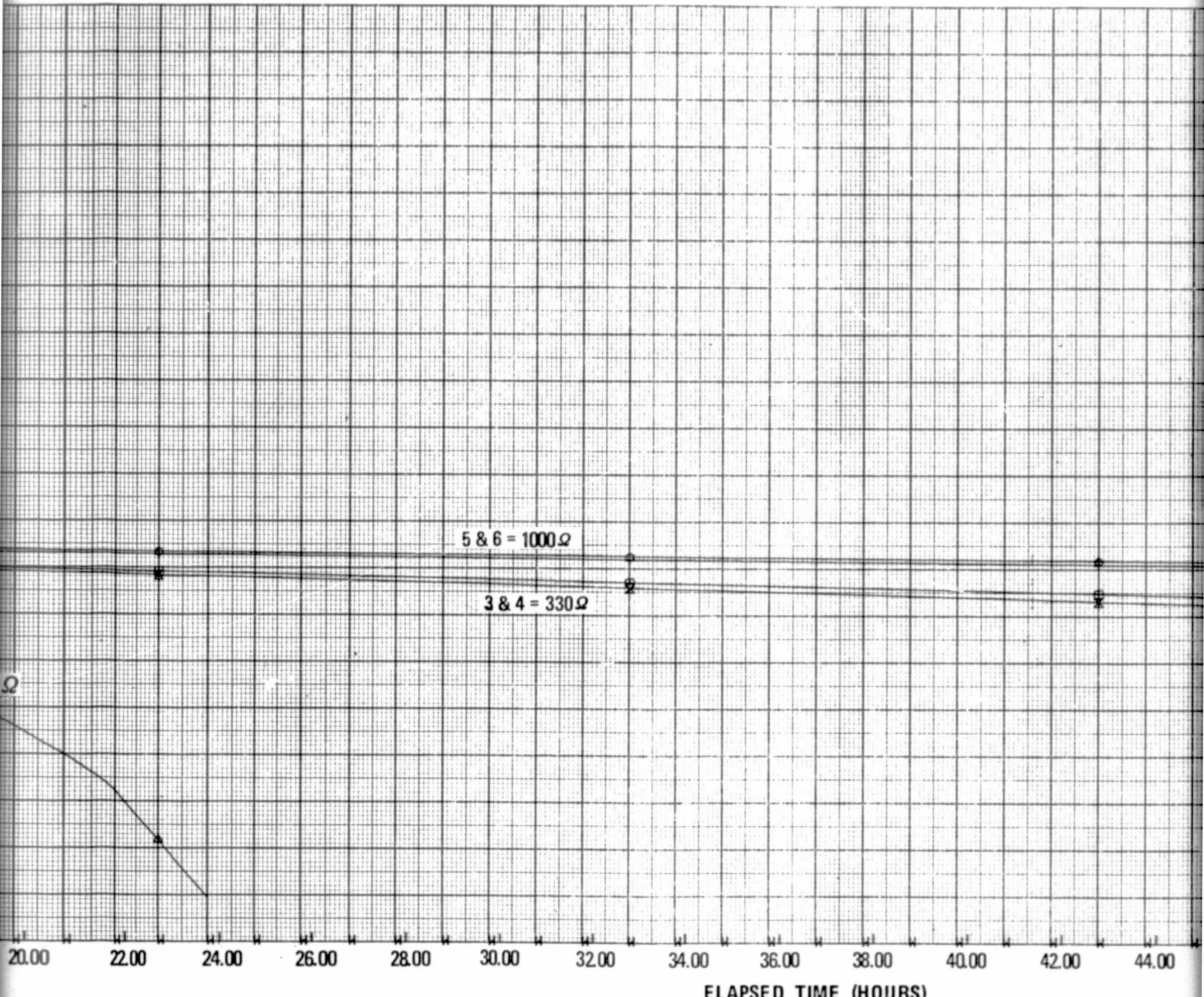

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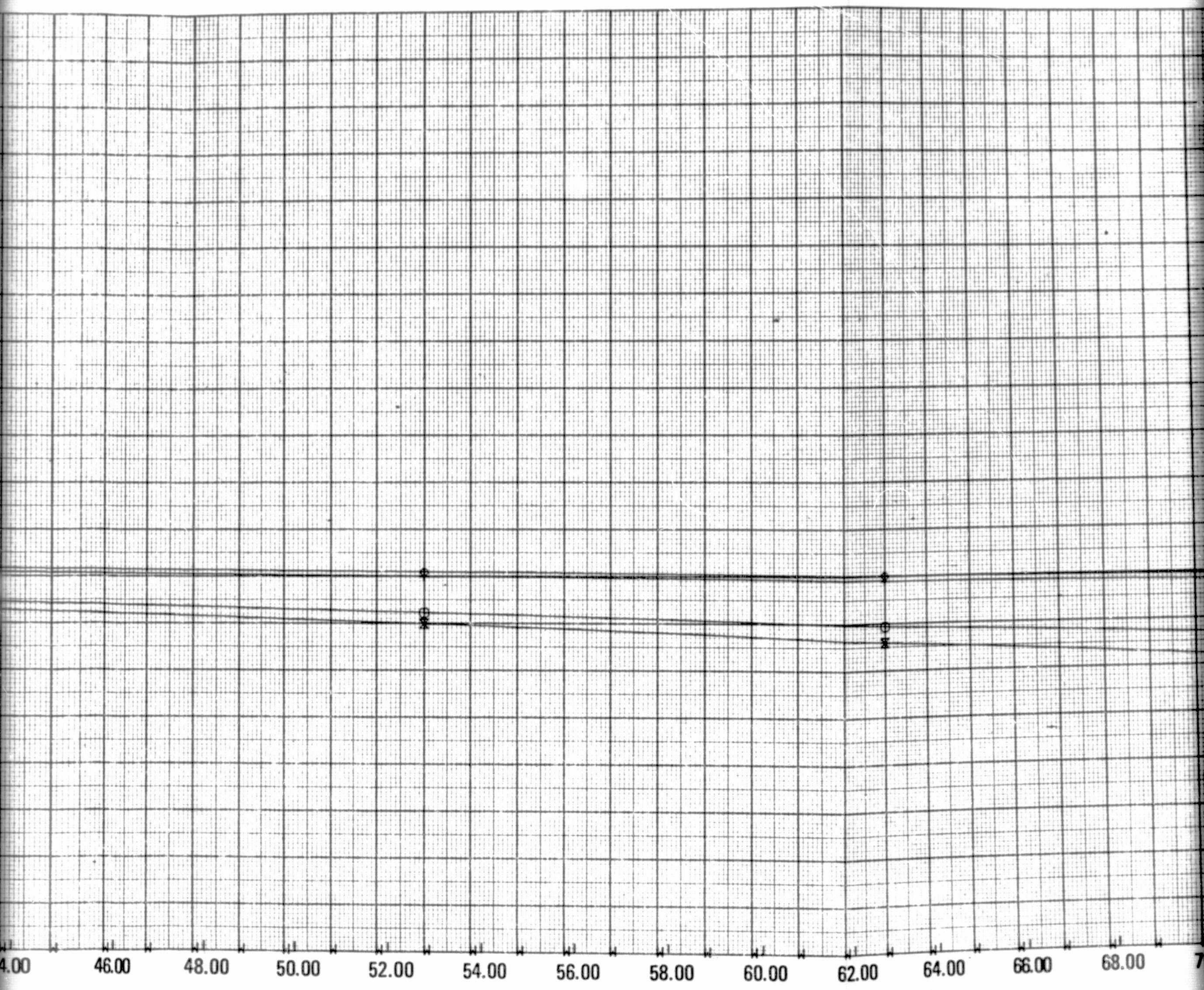

Figure 24. Voltage
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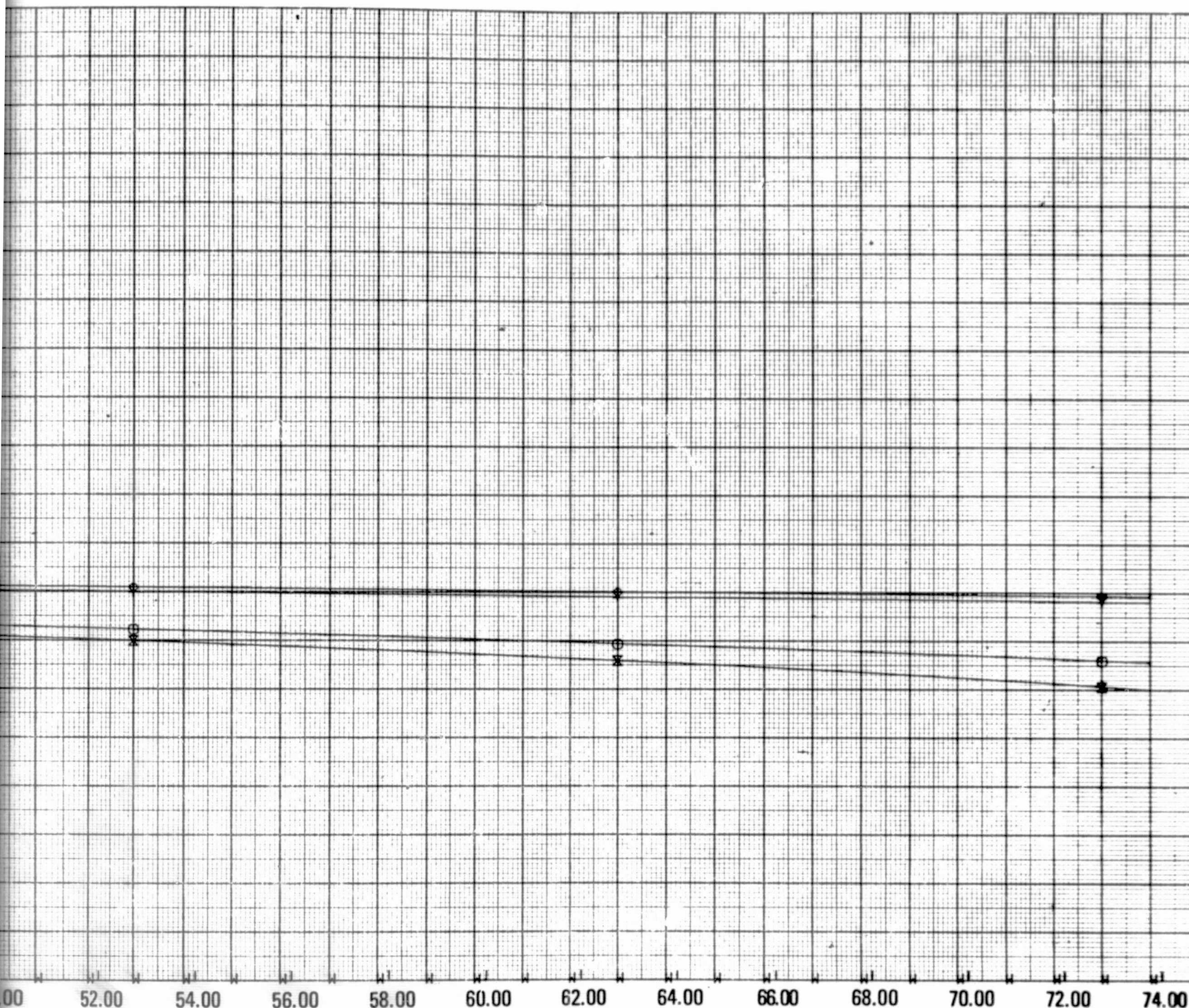

Figure 24. Voltage Decay Data; Test Sequence No. 1, Subgroup 2A After Acceptance Testing

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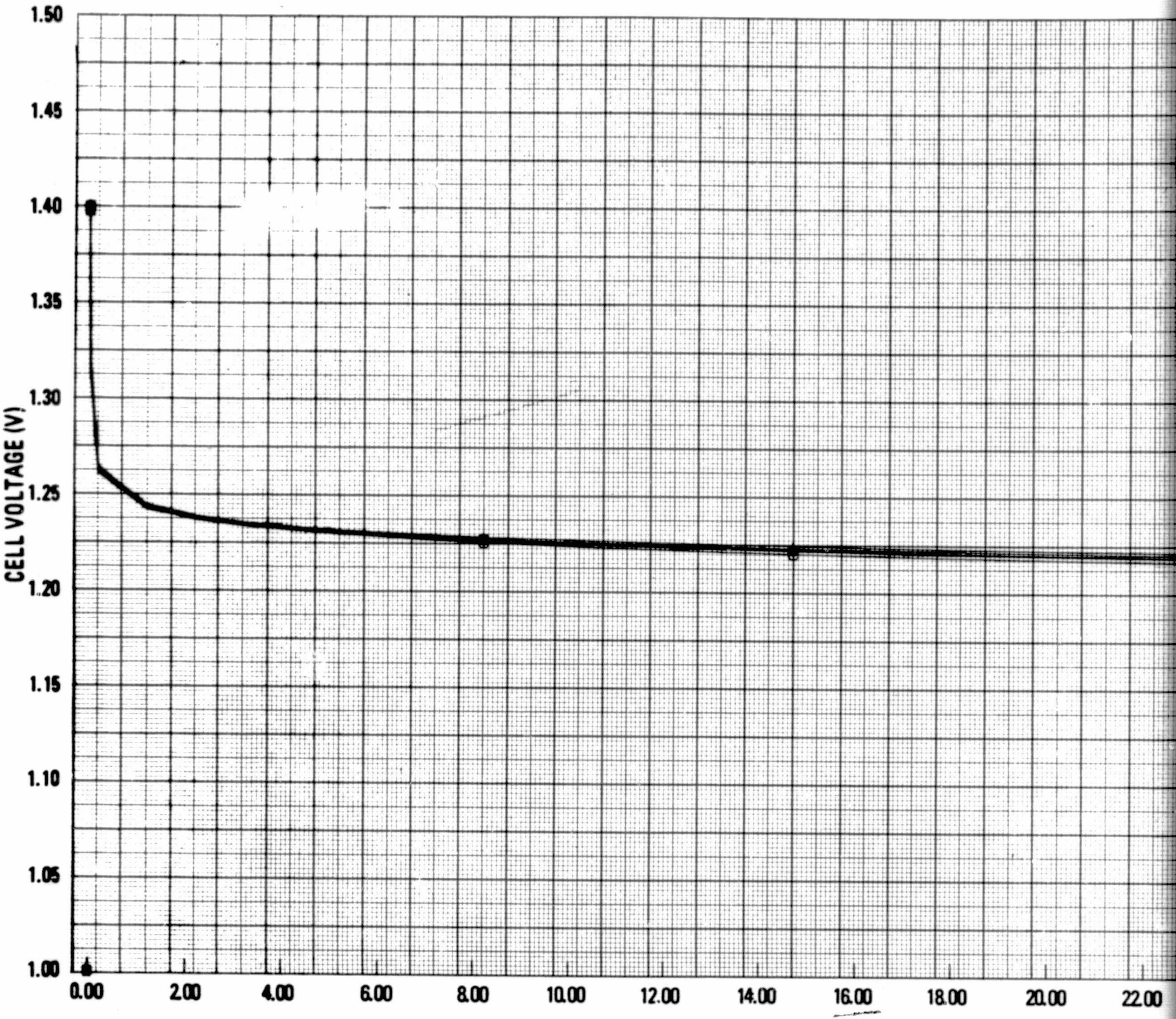

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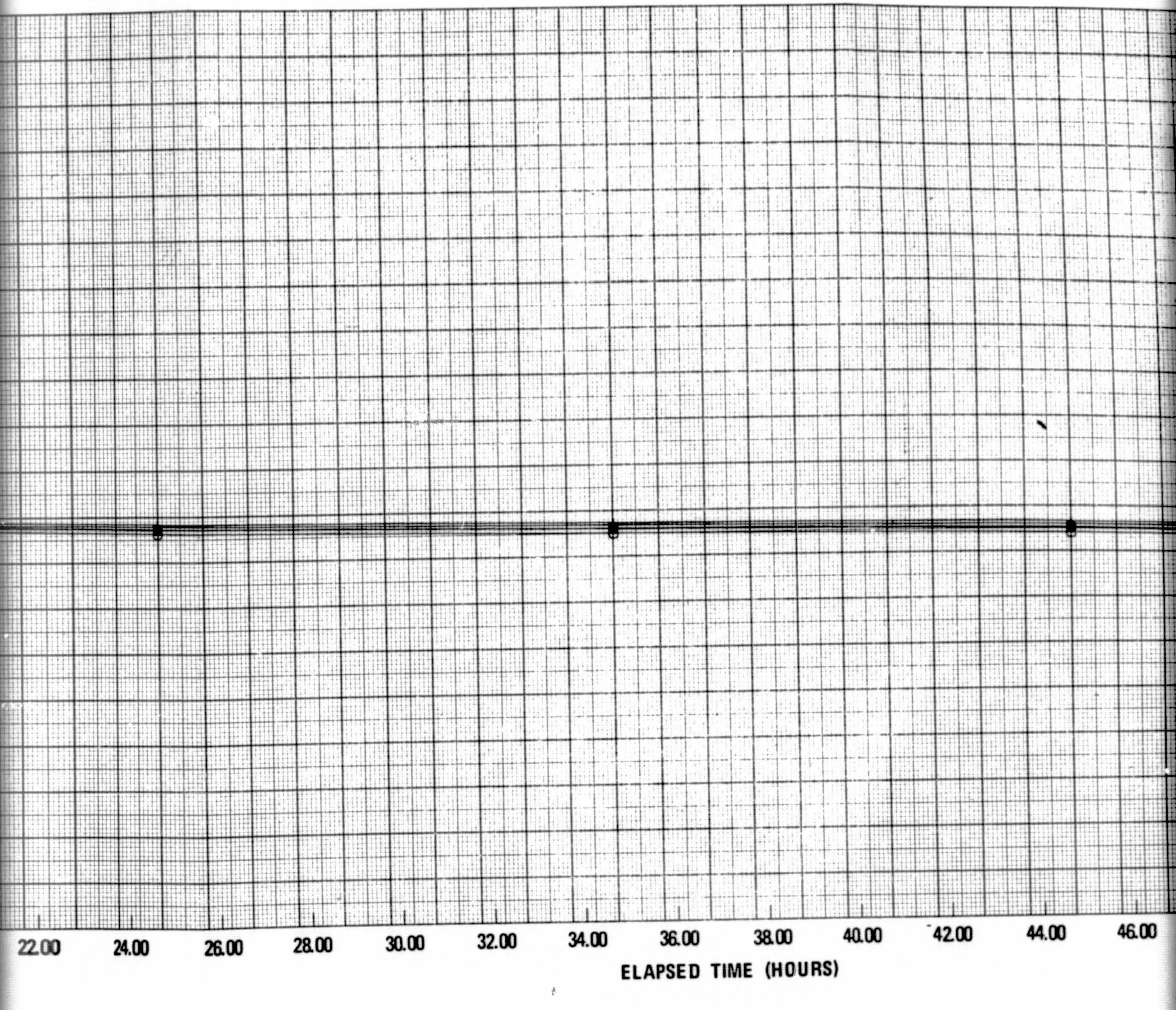

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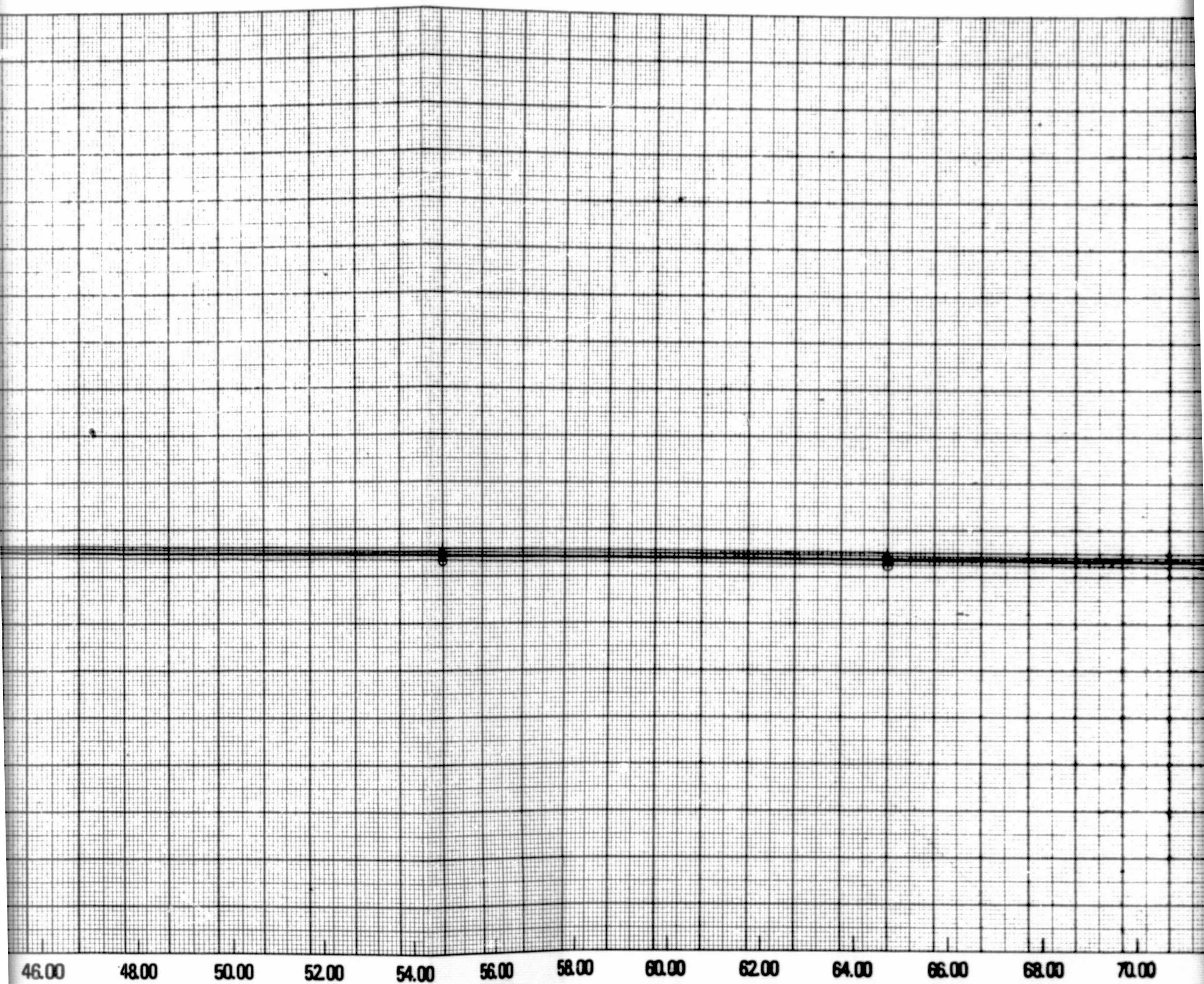

Figure 25. Voltage Decay Data, Test Sequence No. 1, Subgroup 2D After Acceptance Testing

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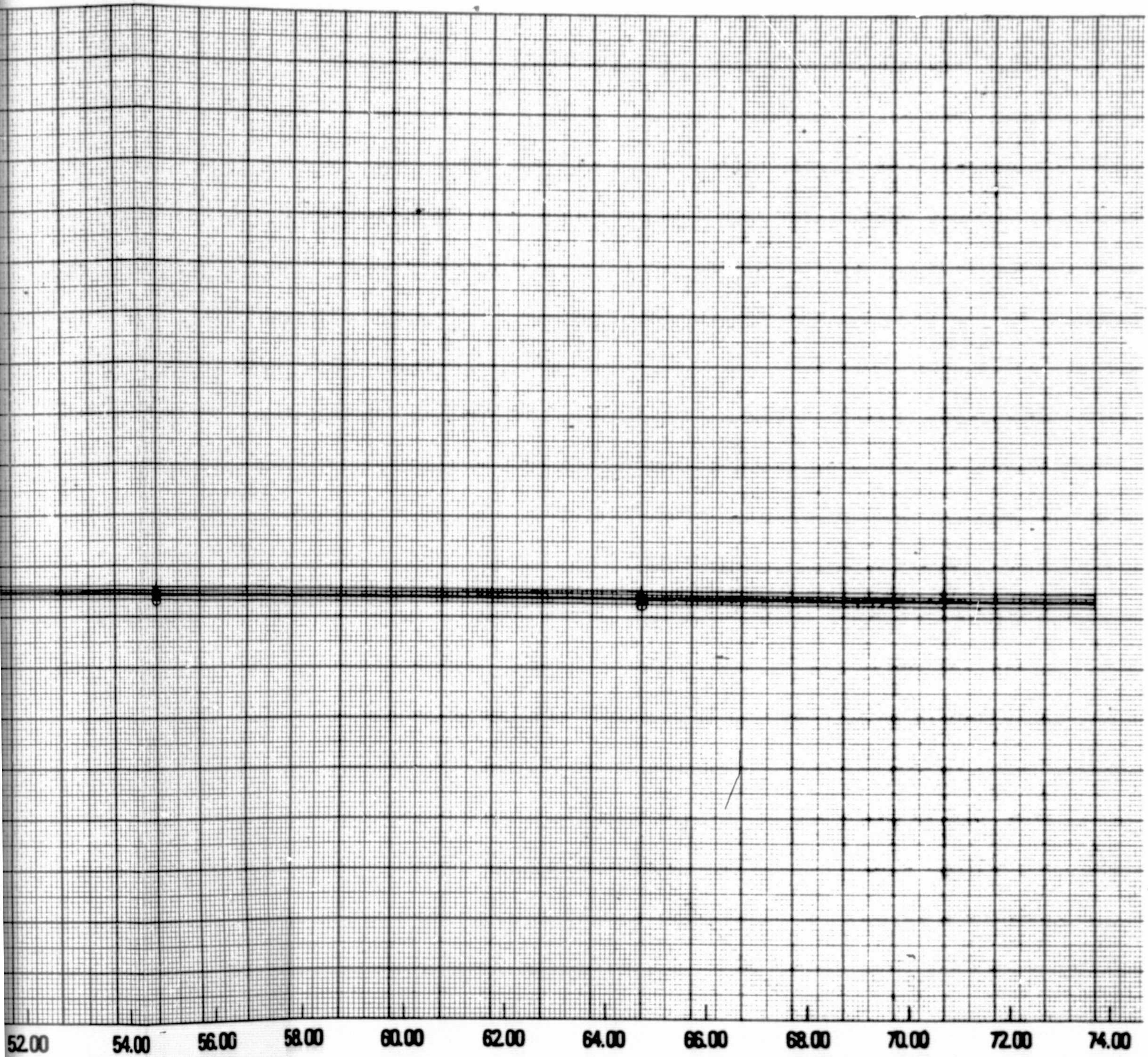

Figure 25. Voltage Decay Data, Test Sequence No. 1, Subgroup 2D After Acceptance Testing

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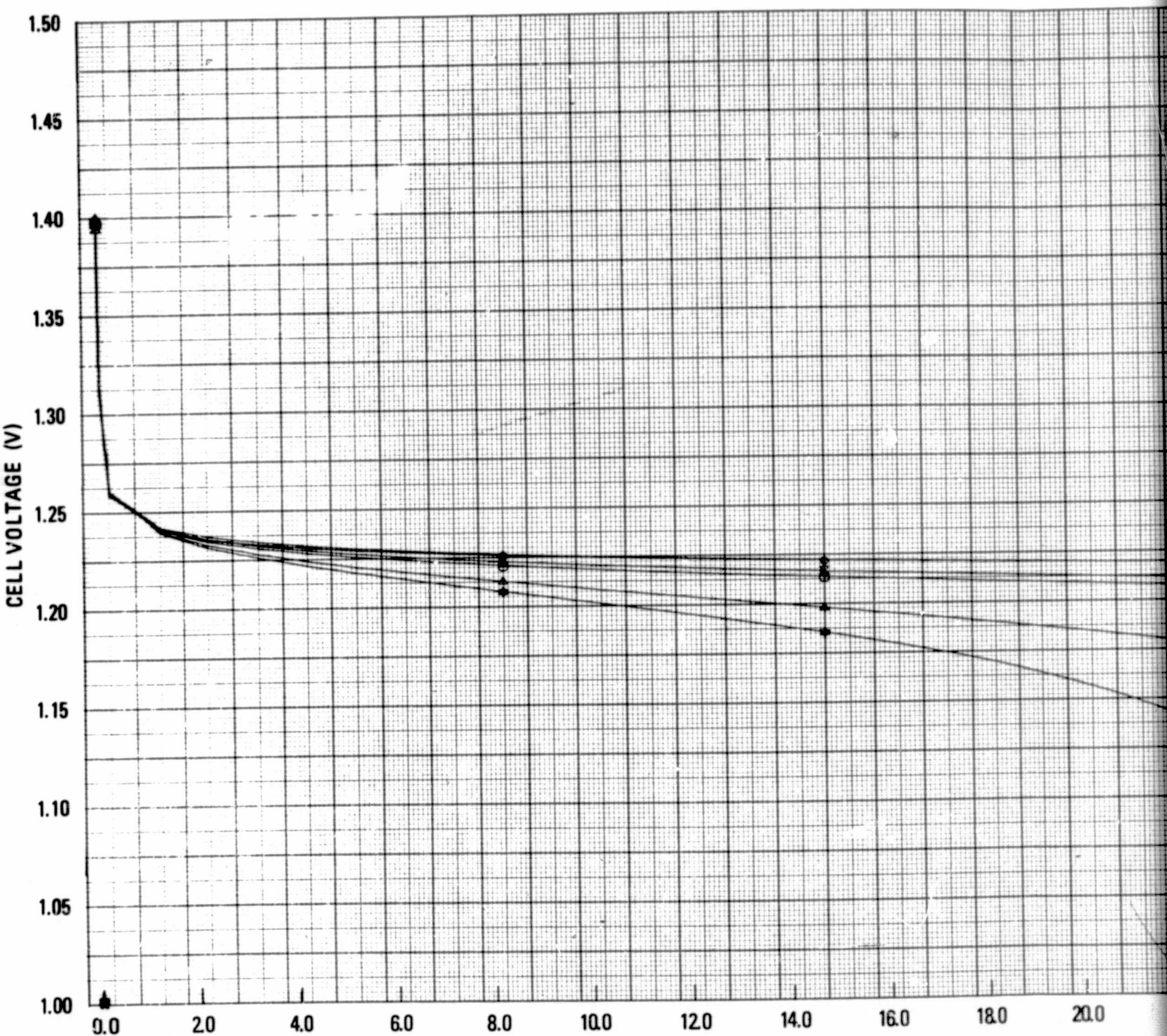

1.50
1.45
1.40
1.35
1.30
1.25
1.20
1.15
1.10
1.05
1.00
CELL VOLTAGE (V)
0.0
2.0
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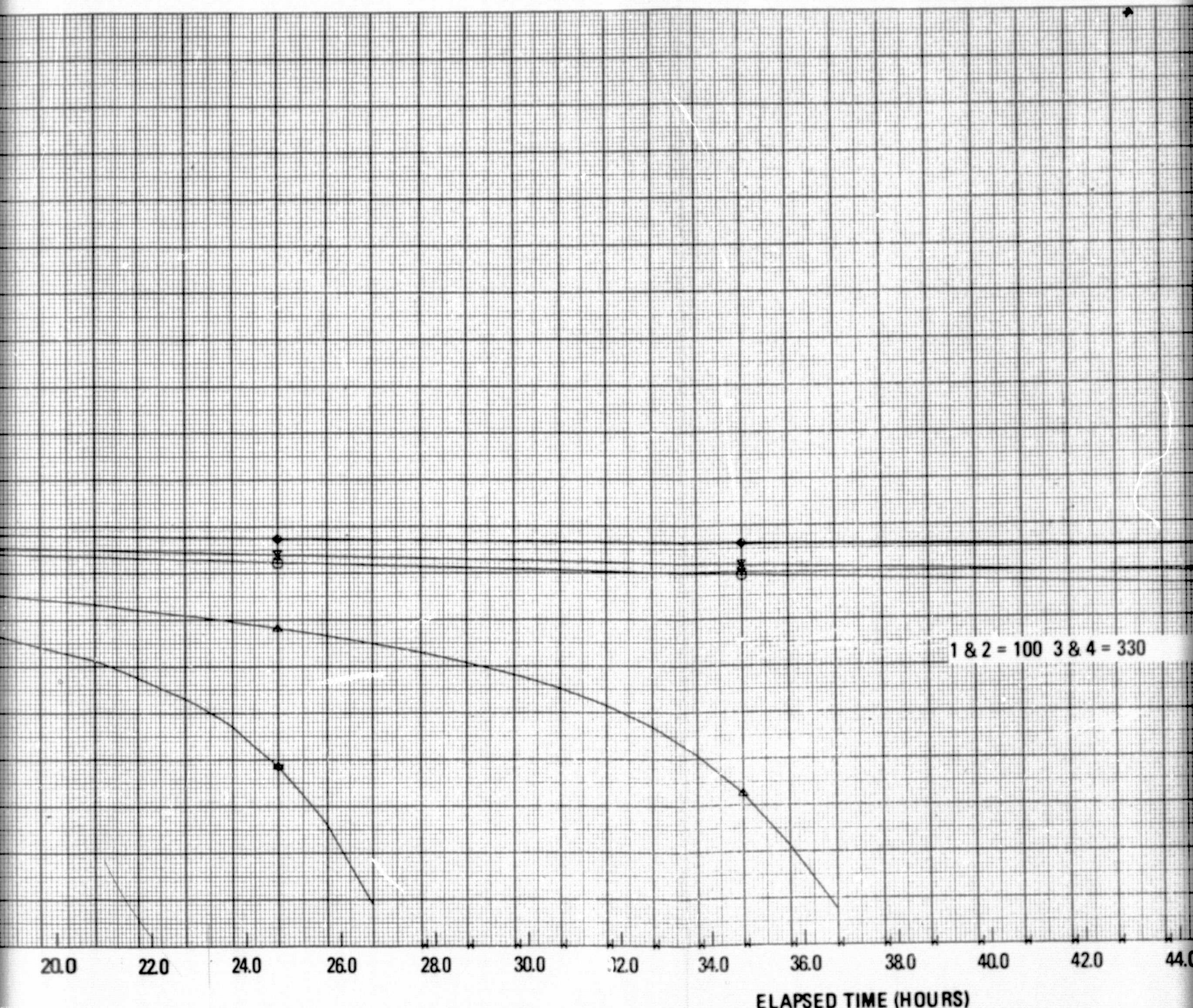
6.0
8.0
10.0
12.0
14.0
16.0
18.0
20.0



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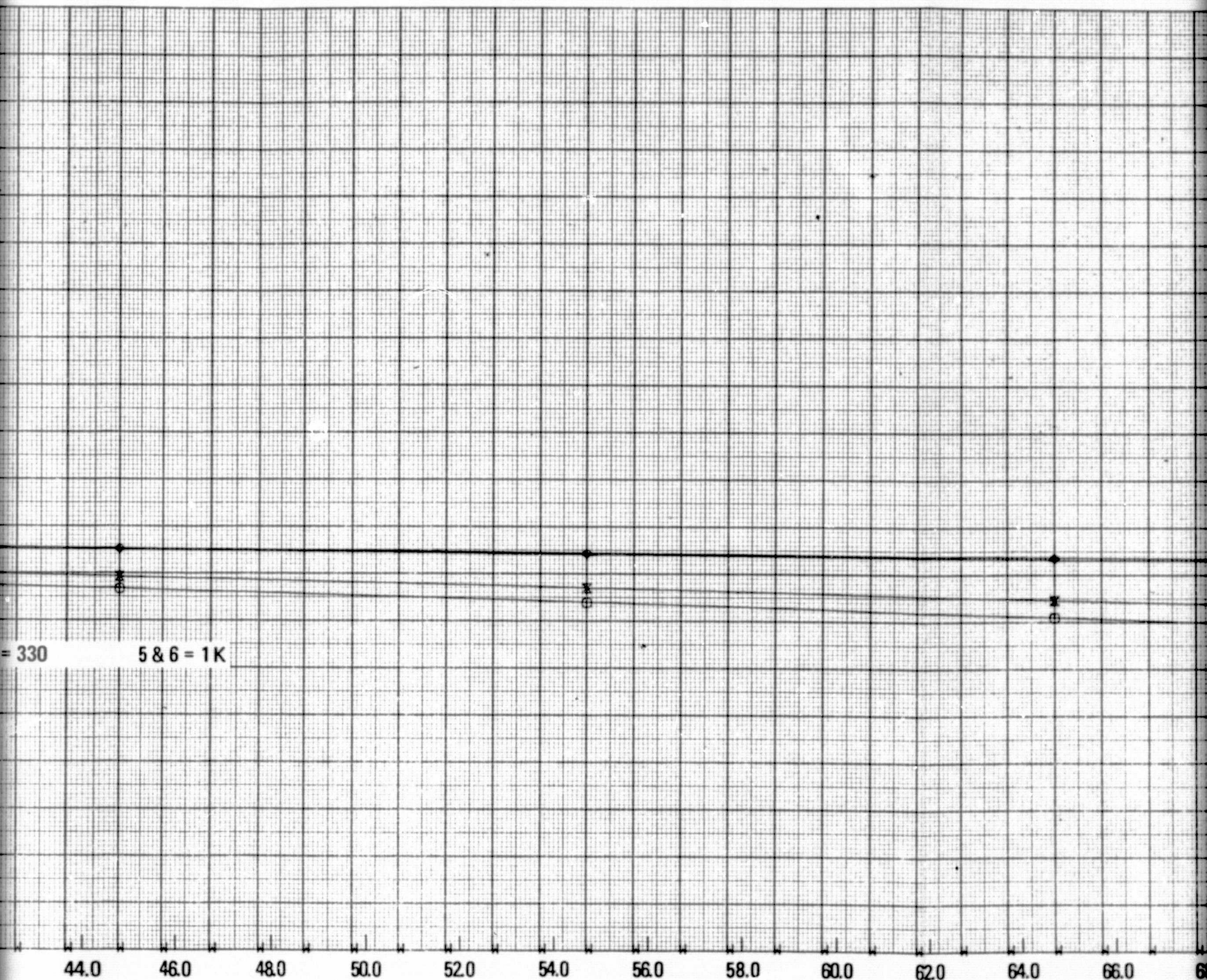

Figure 26. V
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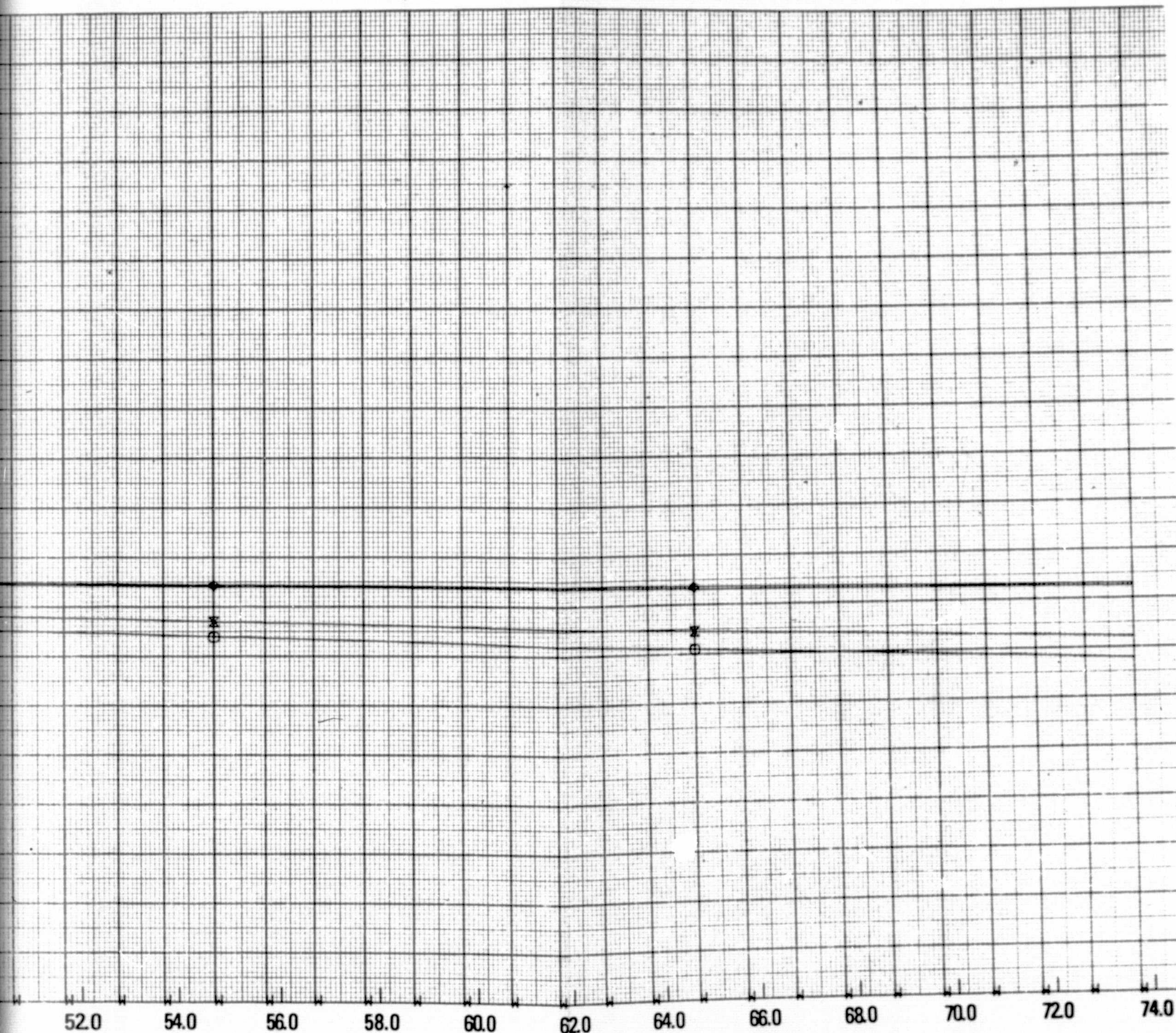

Figure 26. Voltage Decay Data, Test Sequence No. 1, Subgroup 2C After Acceptance Testing

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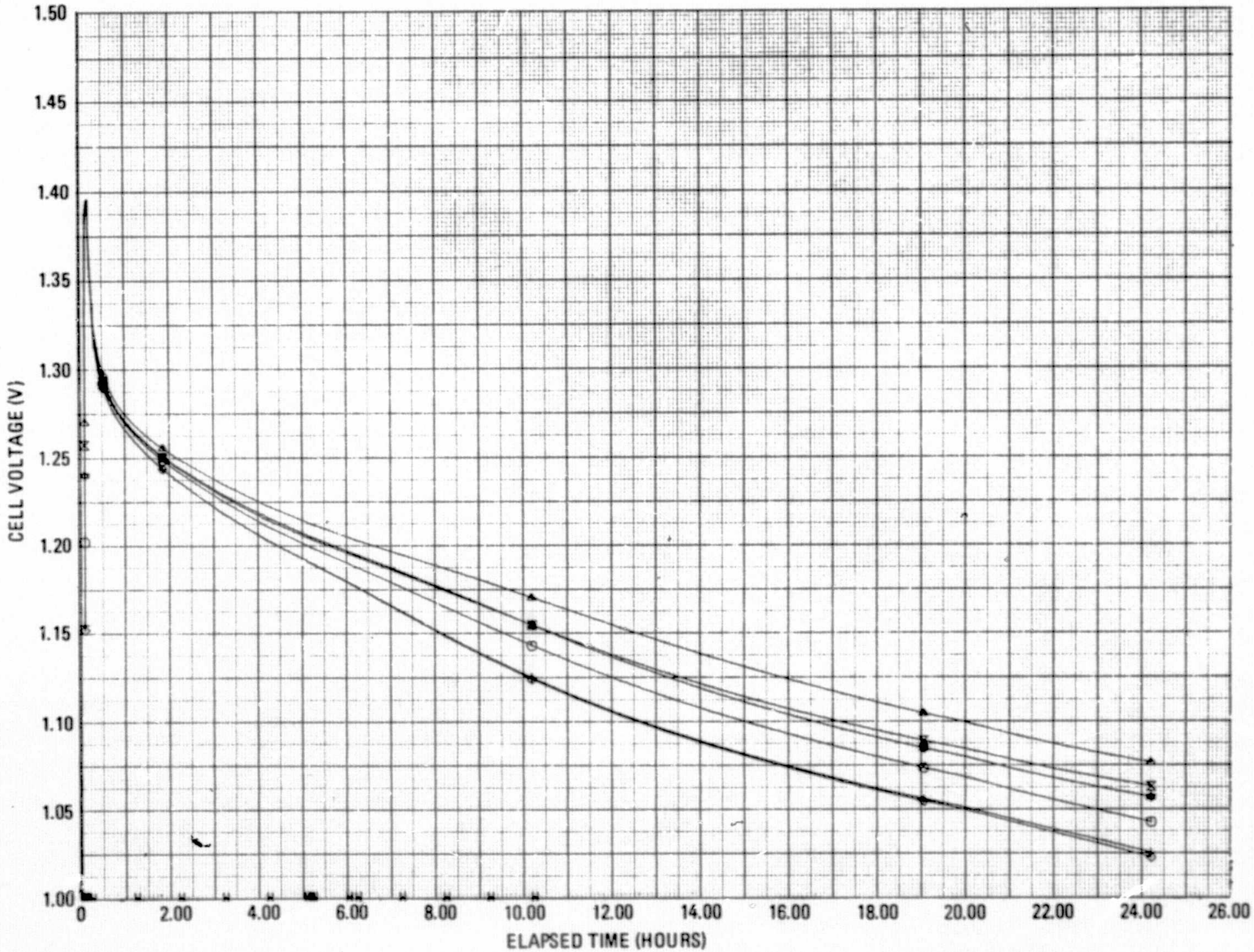


Figure 27. Voltage Decay Data, Test Sequence No. 2, First Open-Circuit Stand

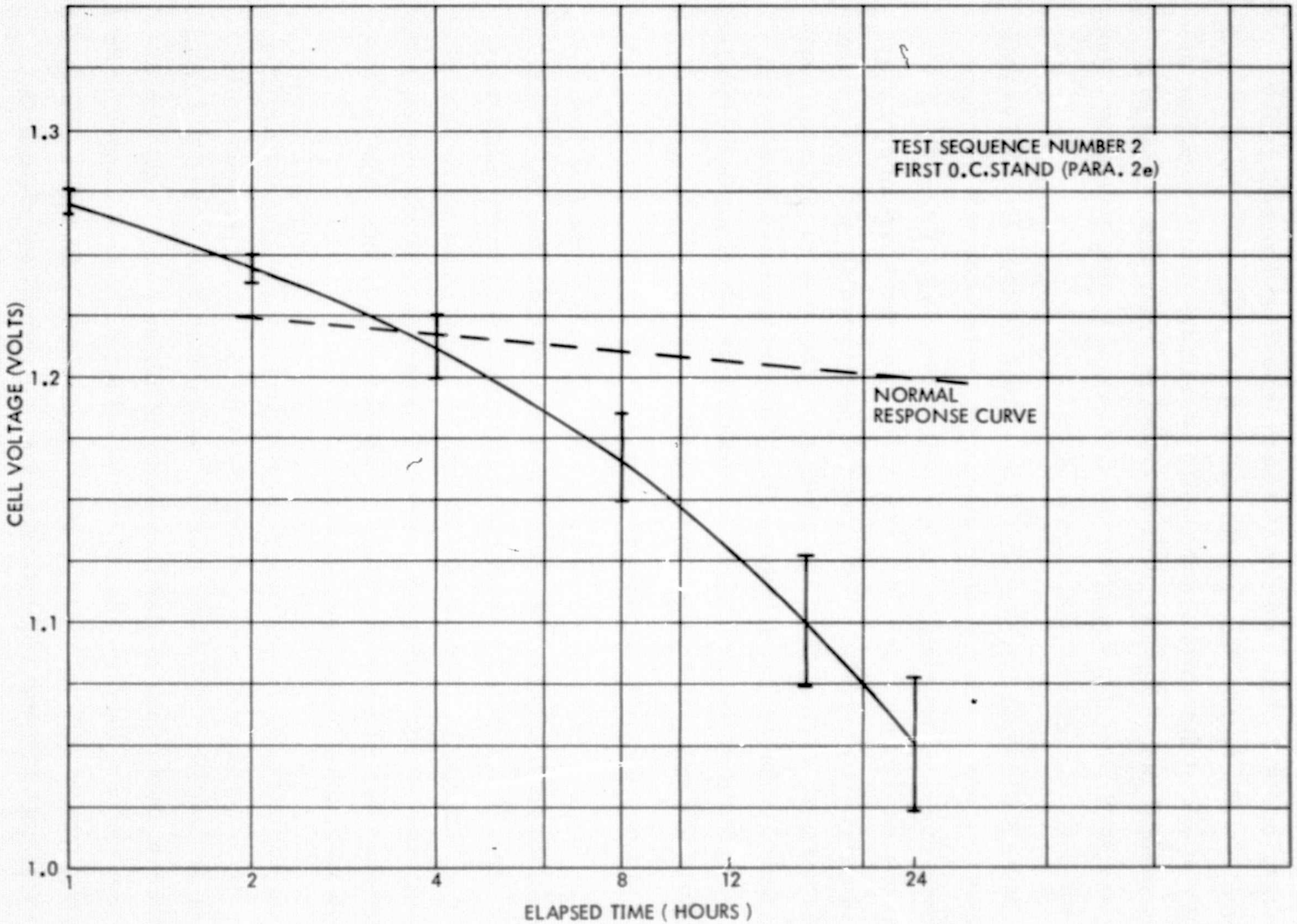


Figure 28. Semi-log Plot of Data from Figure 27

After the rapid voltage decline described above, the cells were subjected to a complete Voltage Recovery Test, including a full charge, discharge, and 0.5 ohm load for 16 hours (see Test Procedure for Sequence No. 2, Steps 2(f), (g), (h) and (i)). A plot of the open circuit voltages of six of the cells from a point 30 minutes after removing the resistors until the end of 24 hours is shown in Figure 29. Note that there was very little change after the first eight hours, when the voltage of all cells were together and over 1.20 volts.

After another short-down for 4 hours the Voltage Decay Test was repeated (without another charge-discharge cycle after the Voltage Recovery Test). The results for six cells receiving a 6 minute C/10 charge are shown in Figure 30. The curves for the 12 minute charge were identical in shape but 20 mV higher than those shown. The spread was the same in each case (5 mV after 48 hours) thus these older cells responded to open circuit voltage testing in a manner essentially the same as that shown by new cells, and no internal shorting was detected. No further testing was done on this test lot.

4.3.3 Results From Test Sequence No. 3

The cells used for Test Sequence No. 3 were about five years old, and had been on continuous shorted storage for about four out of the five years. The 54 cells in this test lot constituted the largest number of cells of a single type, age-group, and prior history that were available for use on this study. They were used to generate data for use in making comparisons between results from different test variables and to characterize cells after long term storage.

Immediately after removing the shorts for the first time in four years, AC impedances were measured (at 60 Hz) on 12 sample cells selected at random. This was done to indicate whether there had been any adverse electrolyte redistribution resulting in a dry separator condition and hence a high impedance. The values measured ranged from 2.5 to 3.8 milliohms with an average of 3.1 milliohms. As this is about the value measured for new cells of this size, no significant change in impedance had occurred during storage.

At the end of the first Voltage Recovery test on Group 1 (Sequence No. 3, para. 4.1(a) through (e)), the cell voltage range of 17 cells out

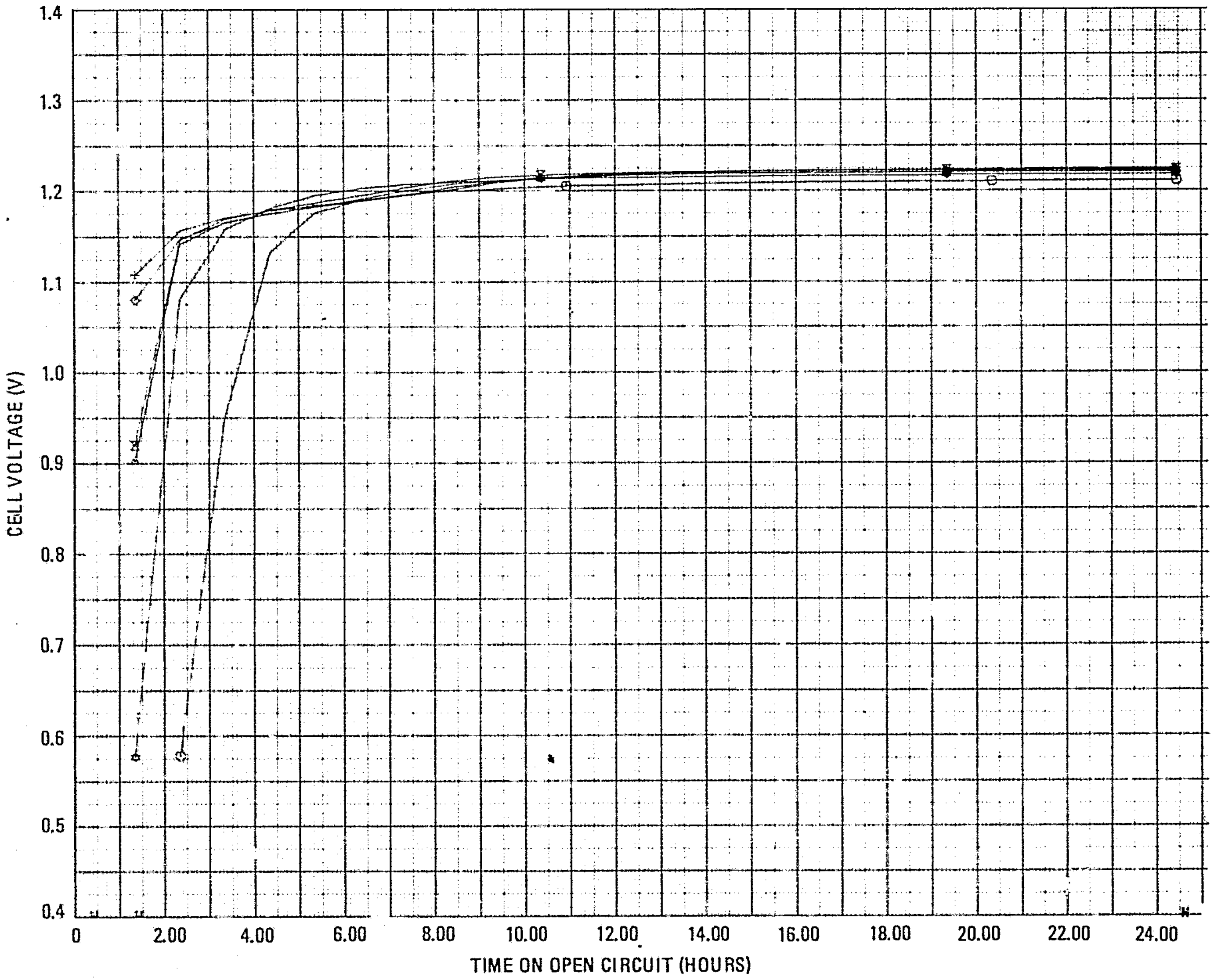


Figure 29. Voltage Recovery Data, Test Sequence No. 2 Para. 2(j), Voltage Decay Test (Six Cells)

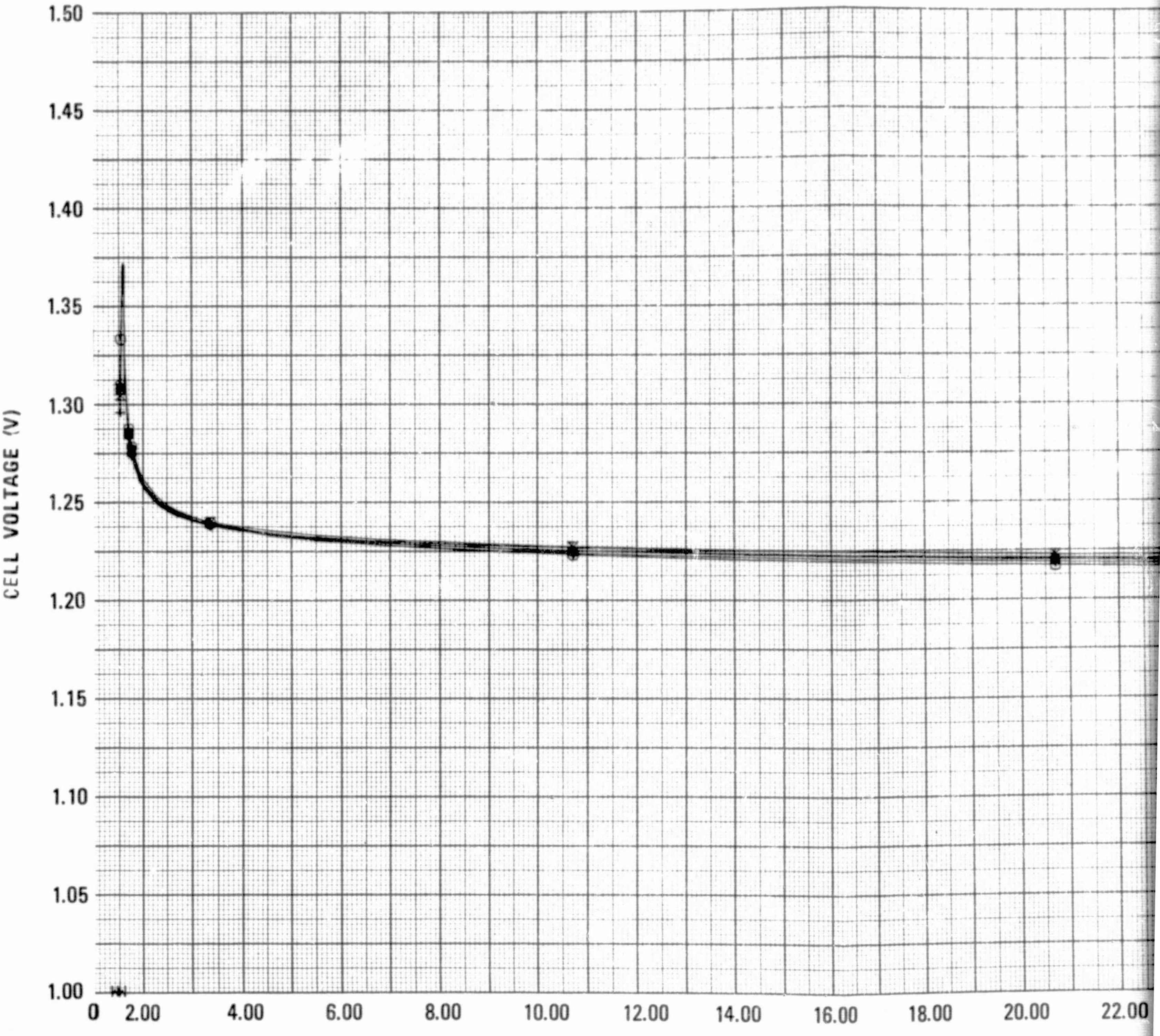
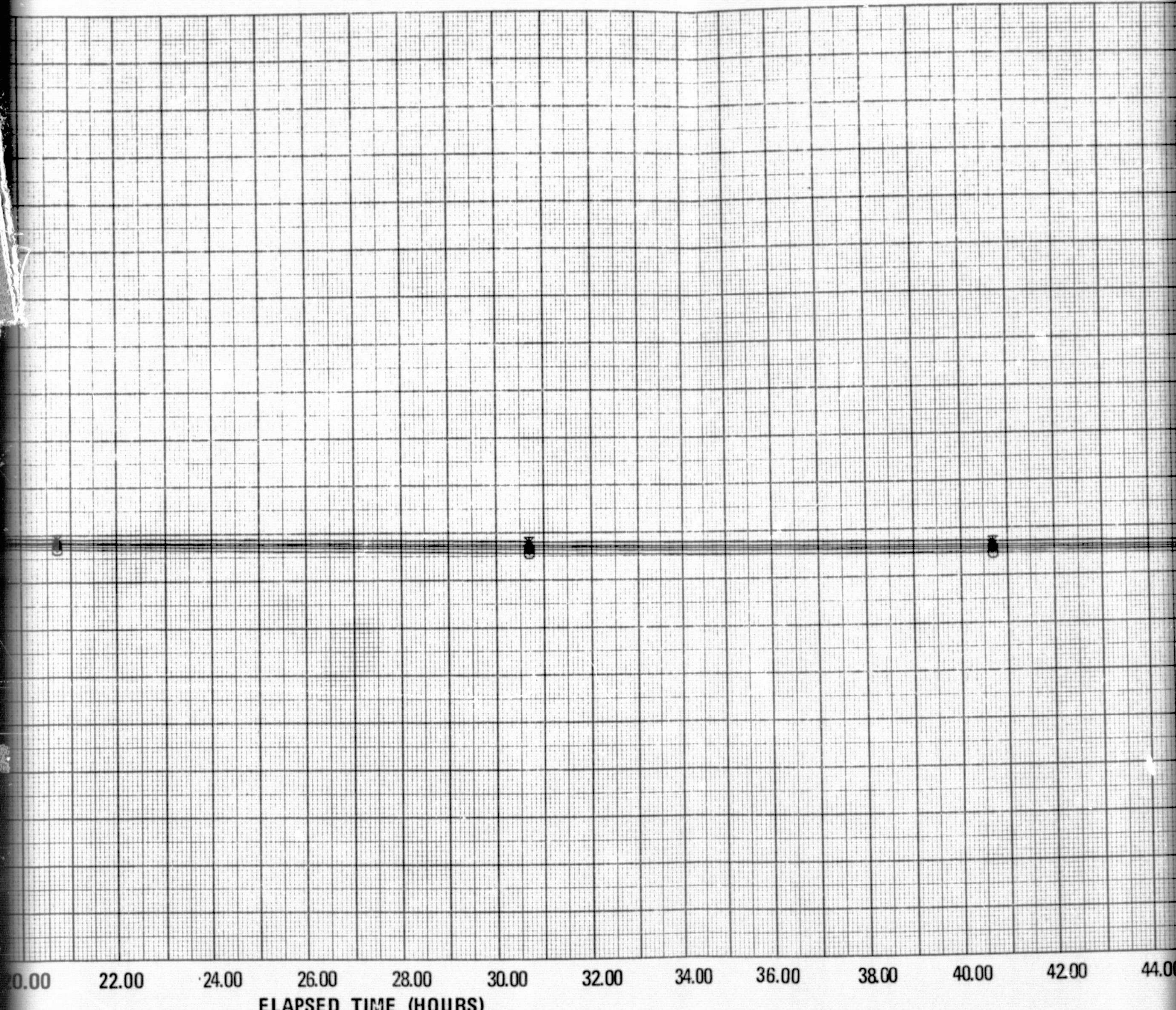


Figure 30. Voltage Decay Data, Test Sequence No. 2, Second Decay Test



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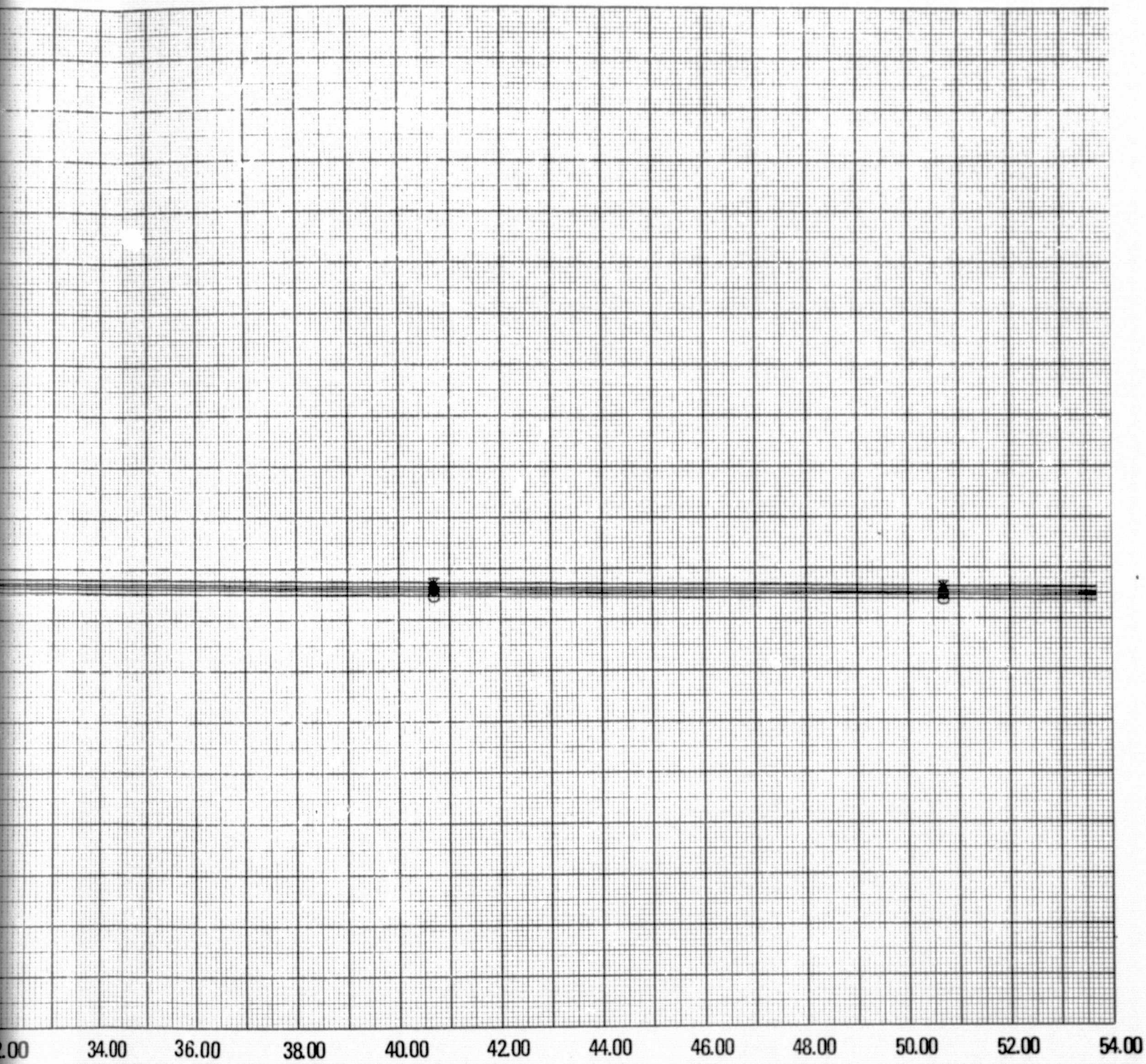

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of 18 was 1.050 to 1.195, and one cell out of 18 (cell No. 1A-3) was at 0.219 volts. The latter cell, which appeared to be internally shorted, was kept in the test group to determine how it would respond to the other tests.

The following test (Voltage Decay) on Group 1 was performed without performing an intervening charge-discharge cycle (Procedure Para. 4.2(h)). The resulting voltage data is plotted versus log of stand time in Figure 31. The vertical bars show the spread in voltages of the six cells in any group. The plots on a linear time scale are omitted here and for most subsequently reported tests because they all appear similar and do not lend themselves to analysis as well as do the semi-log plots.

The voltages for subgroup 1B and 1C, charged for 6 and 9 minutes at the C/10 rate, respectively, fall on straight lines over the period from 2 to 48 hours, whereas voltages for subgroup 1A, charged for 3 minutes, describe an S-shaped curve starting at a significantly lower voltage. Cell 1A-3 remained below 0.7 volt throughout this test, and thus does not appear in Figure 31. Cell 1A-2, which was the lowest of the remaining five cells in Groups 1A in the prior Voltage Recovery Test, was also the lowest in the Voltage Decay Test. A different cell (1C-6) was lowest in Group 1C.

The effect on the voltage recovery response of adding a 24 hour shorted period to the usual 16 hour resistive discharge was then determined with the Group 1 cells. At the same time two of the three subgroups in Group 1 had resistors attached to obtain sensitivity data, (Procedure Para. 4.2(n)). The following data represent the results:

Subgroup	Resistance Attached (ohms)	Max. Voltage (in Volts) After 1 Hour	24 Hours	48 Hours
1A	None	0.173	0.690	1.033
1B	500	0.167	0.248	0.245
1C	1000	0.154	0.717	0.809

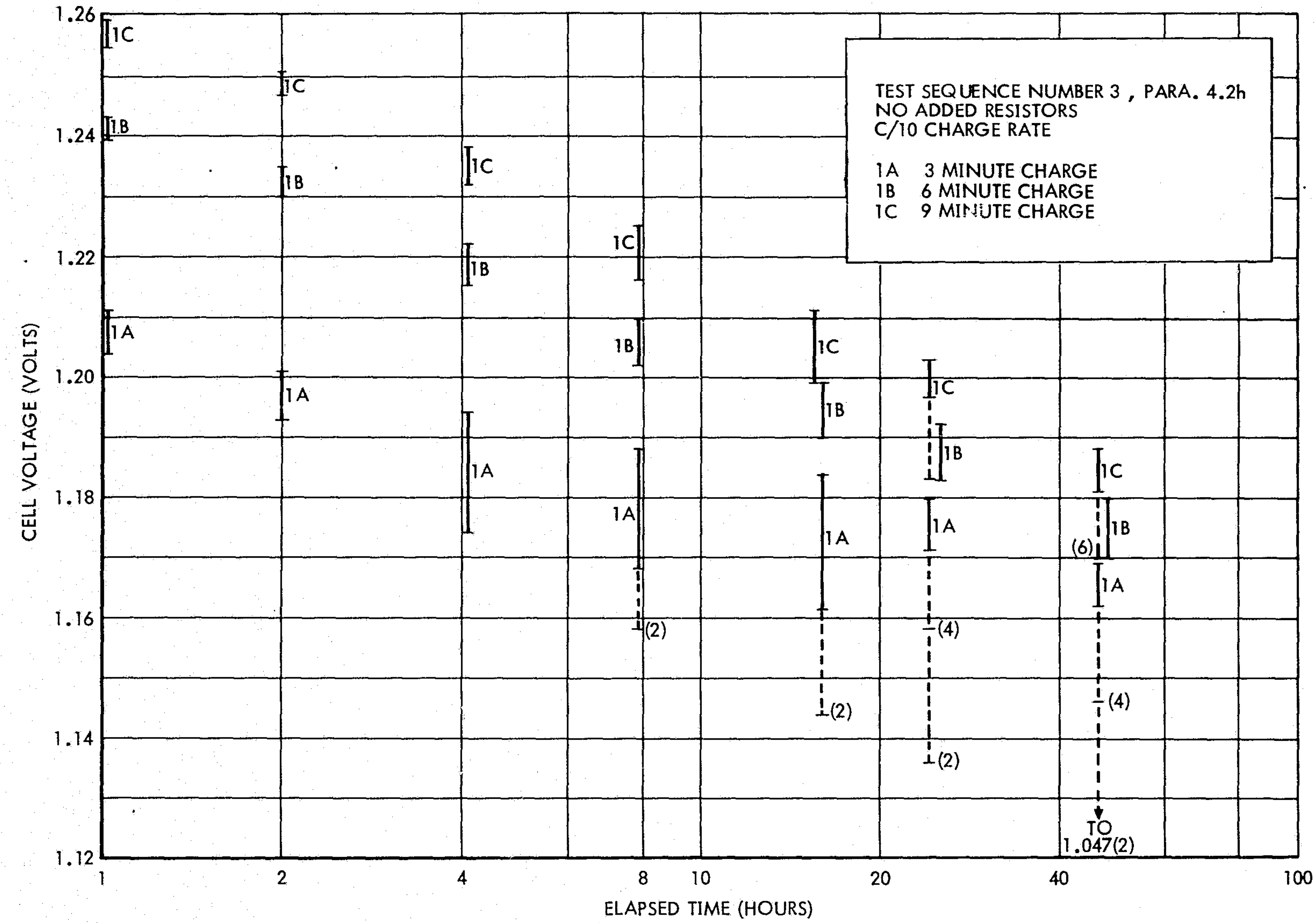

Figure 31. Voltage Decay Data, Test Sequence No. 3, Paragraph 4. 2(h)

The voltages at 24 hours on this test for subgroup 1A (no resistors attached) were far lower than at 24 hours on the first voltage recovery test for this subgroup (also no resistors). Thus the results of this type of test appear to be quite sensitive to the degree of discharging on a low resistance load that precedes the open circuit period. In view of the results described above for these cells, these voltages cannot be the result of internal shorts, and hence must be a result of improper conditioning.

The last test in the sequence for Group 1 was a seven day charged stand with resistors on two subgroups out of three (Sequence No. 3, para. 4.2(u)). The results are plotted in Figure 32. Note that the data for Subgroups 1A and 1C (no resistor and 1000 ohms, respectively) are essentially coincident, whereas the data for Subgroup 1B (500 ohms) is distinctly lower. The slope of the trend lines is the same for all three subgroups, however. This slope was about constant (straight line versus log time in hours) at 0.05 V per decade (hours) up to 100 hours. Note that Cell No. 1A-3 again shows up clearly as having the lowest voltage.

The results for Groups 2 and 3 on Test Sequence No. 3 will be presented together. The first Voltage Decay Test on these two groups compared responses after 16 hours on a resistor plus 4 hours shorted with those after 16 hours on a resistor plus 24 hours shorted (Procedure Para.'s 4.3(f) and 4.4(f)). The voltage data is shown plotted in Figures 33 and 34. Note that the voltage scale is displaced by 30 mV between these two plots.

Voltages from Group 3 cells were 20-30 mV lower at 24 hours than those of the corresponding subgroups in Group 2, and the slopes of the trend lines for Group 3 were about 10 mV per decade (hours) greater (more negative). Subgroup 2A (charged for only 3 mintues at C/10) showed cells that were 10 to 15 mV below the subgroup average while the voltages of subgroup 3A cells (charged 3 minutes) were scattered over a 50 to 100 mV range.

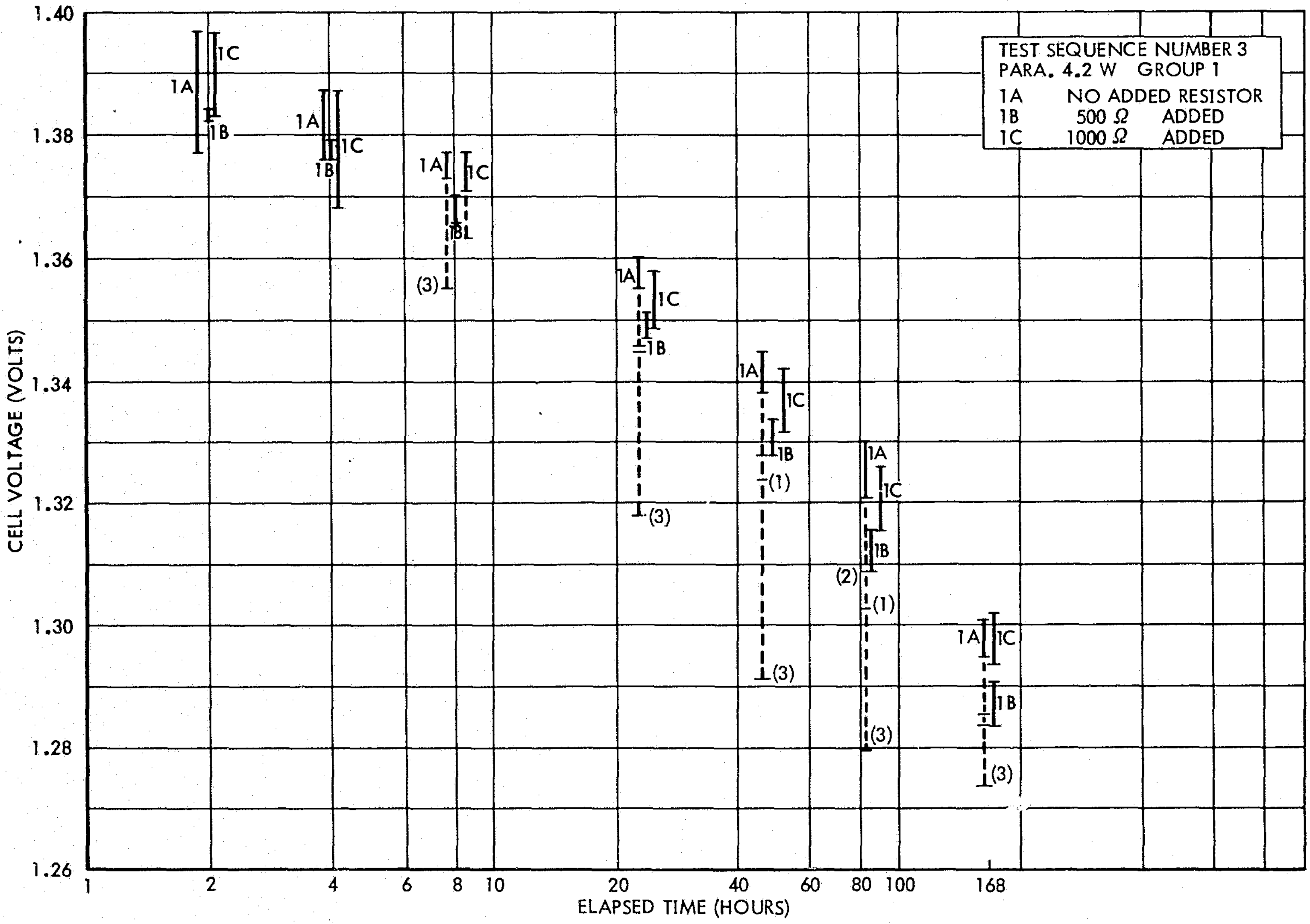

Figure 32. Seven-Day Charged Stand Data, Test Sequence No. 3, Paragraph 4.2(w)

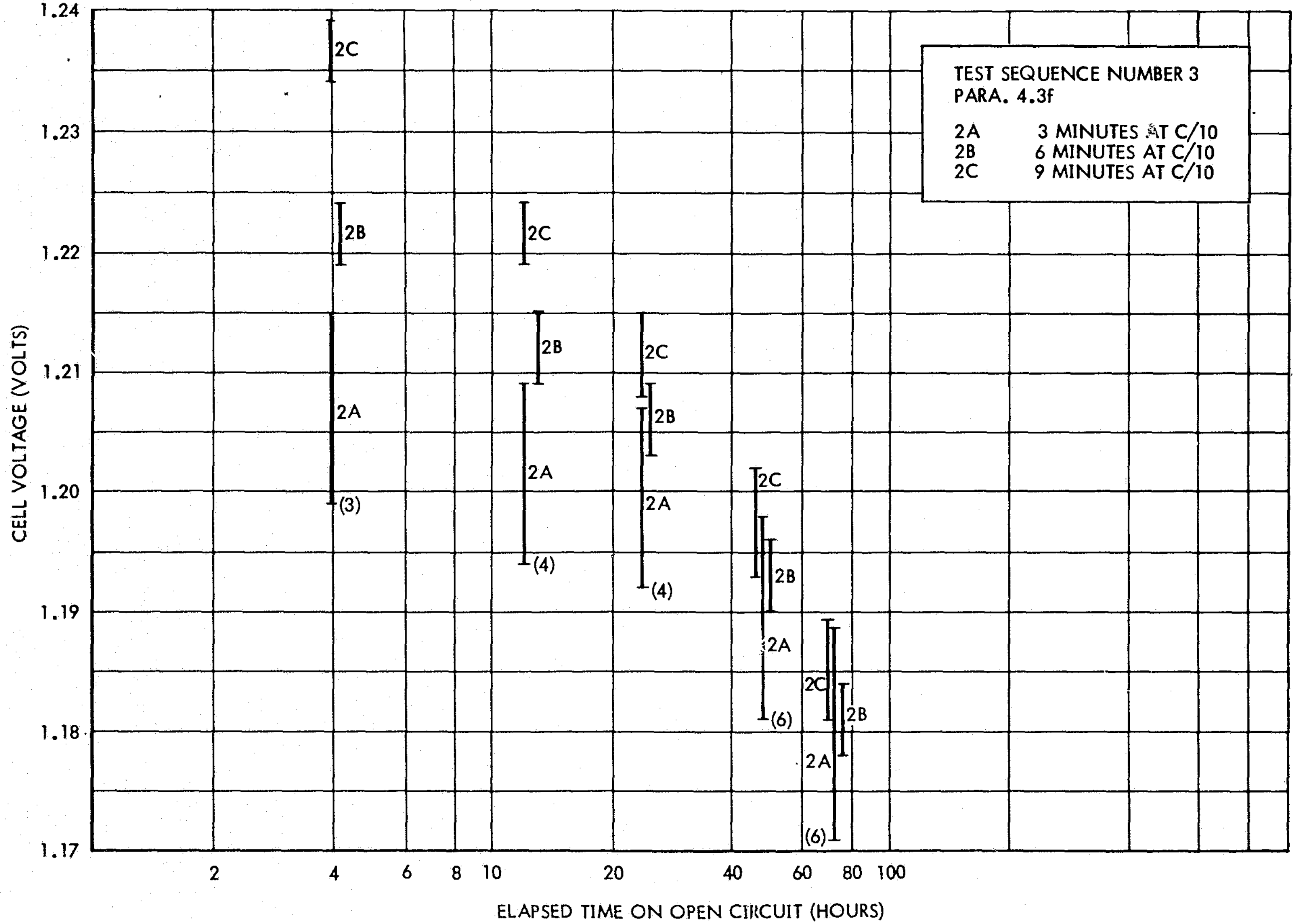

Figure 33. Voltage Decay Data, Test Sequence No. 3, Paragraph 4.3(f)

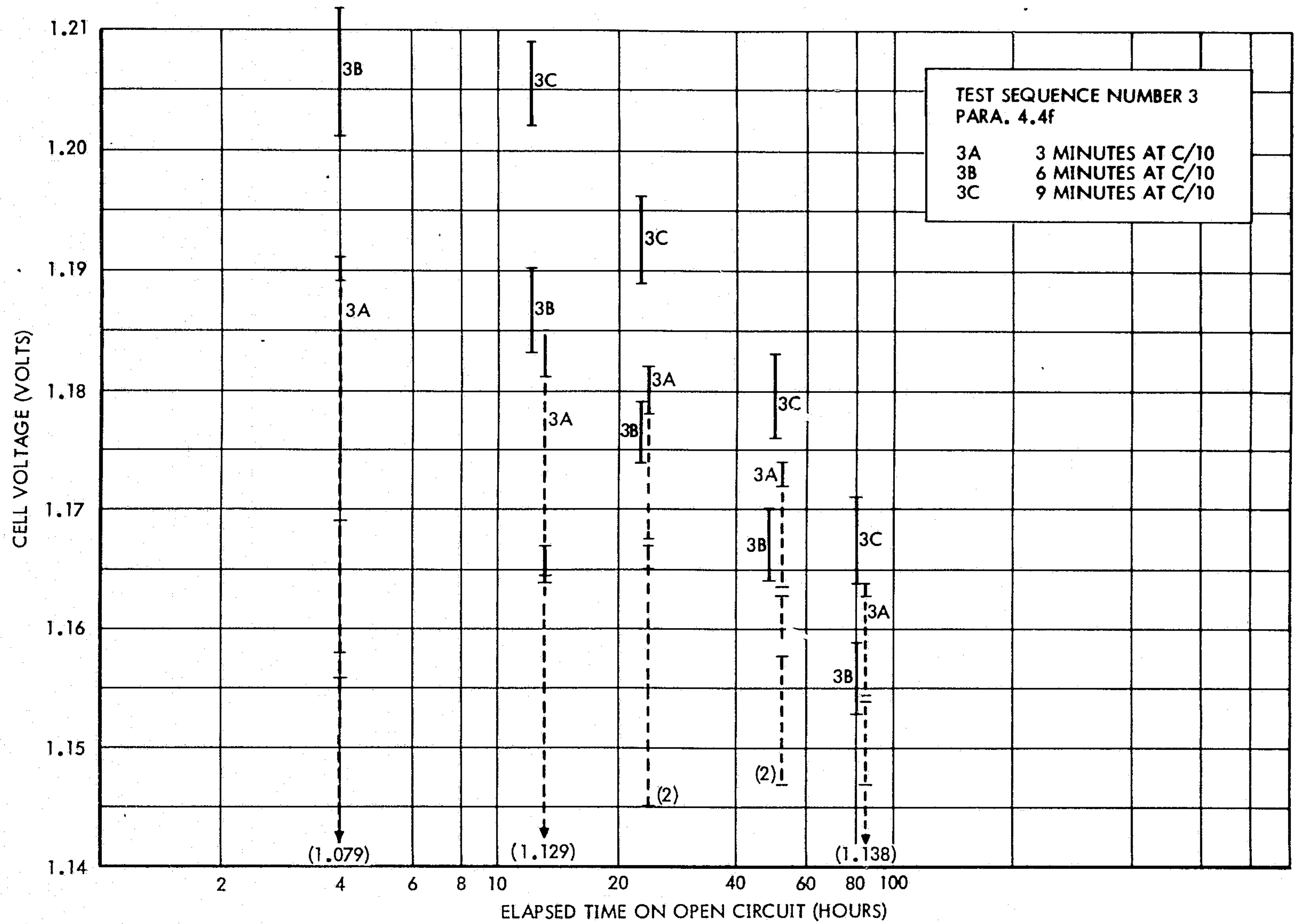

Figure 34. Voltage Decay Data, Test Sequence No. 3, Paragraph 4.4(f)

These results indicate an interdependence of the short-down time prior to the brief charge, and the amount of charge introduced. A three minute C/10 charge was marginal after 16 hours on 0.5 ohm plus 4 hours shorted, and a 6 minute charge is probably the minimum required after a total of 40 hours on 0.5 ohm with or without the use of a dead short for part of that time.

The results of the following Voltage Decay Test (Procedure Paragraph 4.3(h), 4.3(n), 4.4(h) and 4.4(n)), wherein all cells were given a 6 minute C/10 charge are shown plotted together in Figure 35. Note that four of the six groups had resistors attached during the stand period. It may be seen that under these conditions the effect of a 2000 ohm resistor (on 2A and 3B) was noticeable only in one of the two subgroups (2A) and then only after 36 hours, while the effect of 1000 ohms (on 3A) was significant only after 24 hours. By contrast, the effect of the 500 ohm resistors on 2C was obvious after only 8 hours. Thus the usable sensitivity on this test was about 500 ohms for the 15 Ah cells involved.

The final test on Groups 2 and 3, Sequence No. 3, was a 7 day charged stand (paragraph 4.3(w) and 4.4(w)), with resistors on four of the six subgroups. The data is plotted in Figure 36. Note that there is no clean-cut separation by voltage of the cells with different load resistors attached, although subgroup 2C, with the lowest resistance (500 ohms), is at the low end of the distribution. Also there does not appear to be a correlation between the size of the resistance added and the spread of voltages within a subgroup, as there is in the other Voltage Decay Test. This may have been due to the fact that the resistances used were higher than can be clearly detected by this procedure.

The capacities of the cells in Groups 1, 2, and 3 measured before and after the seven day stand are listed in Table 4-2, together with the calculated percent capacity retention. A summary of subgroup average retentions versus resistors added is shown in Table 4-3.

Although there is a slight downward trend of a few percentage points in the retention values as the resistor decreases, the differences are of the same order of magnitude as the repeatability expected for any one subgroup. Hence the usable sensitivity from capacity retention data was about 300 ohms for these cells.

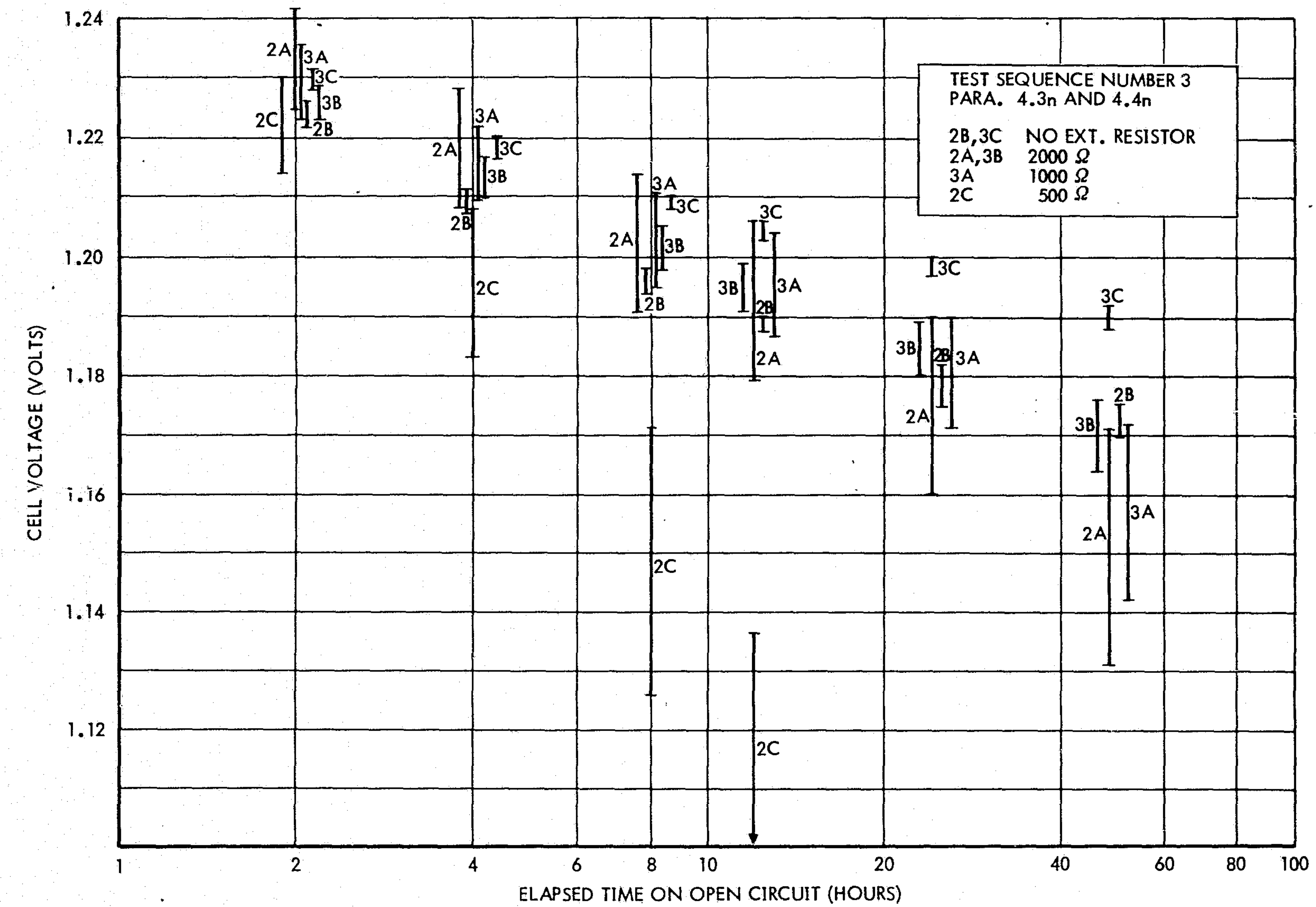

Figure 35. Voltage Decay Data, Test Sequence No. 3, Paragraphs 4.3(h) and 4.4(n)

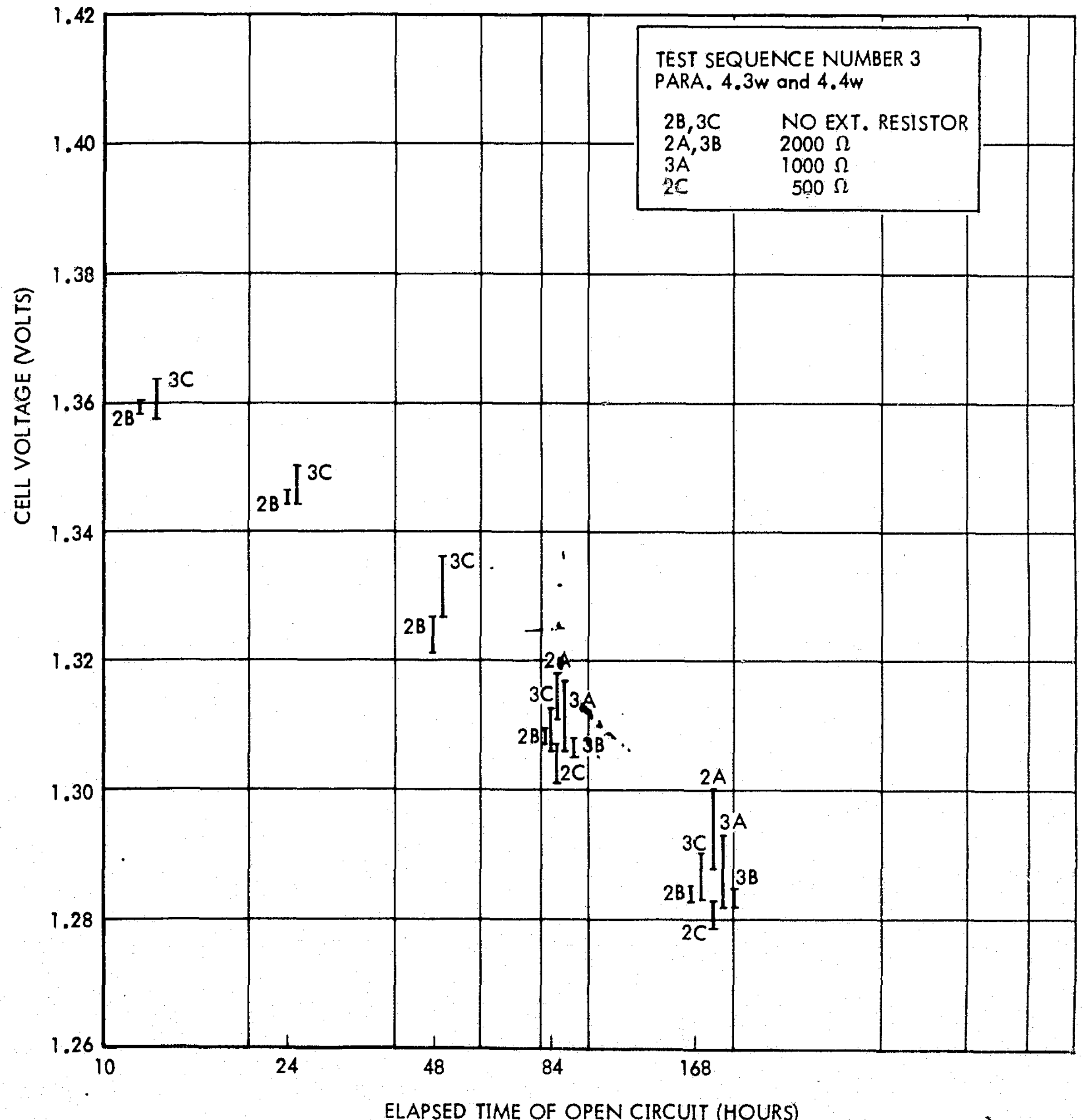

Figure 36. Seven-Day Charged Stand Data, Test Sequence No. 3, Paragraphs 4.3(w) and 4.4(w)

Table 4-2. Capacity Data — Charged Stand Test

Test Sequence No. 3					
		Capacity (Ah)		Capacity Retention (%)	
Subgroup	Cell No.	Before	After	Cell	Subgroup Average
1A	1	20.8	18.00	86.5	
	2	20.0	17.4	87.0	
	3	20.1	14.9	74.1	
	4	20.2	18.34	90.8	
	5	20.5	19.1	93.2	
	6	20.3 (est)	18.92	93.2	90.1(5)*
1B	1	21.2	18.1	85.37	
	2	20.0	17.2	86.0	
	3	21.2	18.3	86.32	
	4	20.2	17.76	87.92	
	5	21.2	17.84	84.15	
	6	20.5	17.67	86.20	86.0(6)*
1C	1	21.2	18.6	87.74	
	2	Not tested	Not tested	--	
	3	21.2	18.4	86.79	
	4	20.8	19.0	91.35	
	5	21.2	17.7	83.49	
	6	21.2	18.6	87.73	87.4(5)*
2A	1	21.6	19.1	88.43	
	2	20.6	18.5	95.37	
	3	20.2	18.14	89.80	
	4	20.5	18.5	90.24	
	5	20.5	18.5	90.24	
	6	20.6	18.0	87.37	90.2(6)*
2B	1	22.0	17.8	80.9	
	2	21.0	18.0	95.4	
	3	20.7	17.6	85.0	
	4	21.2	18.1	85.4	
	5	20.5	17.6	85.9	
	6	21.0	18.1	86.2	86.5(6)*
2C	1	21.8	18.5	84.9	
	2	20.9	17.7	84.7	
	3	21.7	18.2	83.9	
	4	21.0	17.6	83.8	
	5	21.1	17.7	83.9	
	6	21.3	18.0	84.5	84.3(6)*

*The number in parenthesis after the average is the number of cells used to calculate the average value.

Table 4-2. Capacity Data — Charged Stand Test (Continued)

Test Sequence No. 3					
		Capacity (Ah)		Capacity Retention (%)	
Subgroup	Cell No.	Before	After	Cell	Subgroup Average
3A	1	21.1	17.5	82.9	
	2	20.4	17.4	85.3	
	3	19.7	18.2	92.4	
	4	20.6	17.7	85.9	
	5	20.0	16.8	84.0	
	6	20.8	18.3	88.0	86.4(6)*
3B	1	20.8	17.4	83.7	
	2	20.0	17.1	85.5	
	3	20.5	17.3	84.4	
	4	20.4	17.2	84.3	
	5	20.5	17.8	86.8	
	6	20.4	17.3	84.8	84.9(6)*
3C	1	20.5	17.5	85.4	
	2	20.7	17.5	85.3	
	3	21.0	18.2	86.7	
	4	20.6	18.0	87.4	
	5	20.9	18.4	88.0	
	6	20.9	17.8	85.2	86.3(6)*

*The number in parenthesis after the average is the number of cells used to calculate the average value.

Table 4-3. Retention Versus Load Resistance

Subgroup	Resistor Added (ohms)	Average Capacity Retention (percent)
1A	None	90.1
2B	None	86.5
3C	None	86.3
2A	2K	90.2
3B	2K	84.9
1C	1K	87.4
3A	1K	86.4
1B	500	84.3
2C	500	84.3

It is interesting to note that Cell 1A-3, which was very low in voltage on open circuit (Figure 28), showed a clearly lower capacity retention than the subgroup average. Also, Cells 1A-1 and 1A-2, which were more than 10 mV below the average of the highest three cells in Subgroup 1A after the first 24 hours of the 7 day stand, showed capacity losses well below the average loss of the three highest cells in their subgroup.

4.3.4 Results From Test Sequence No. 4

Test Sequence No. 4 involved 12 General Electric 12 ampere-hour cells that were 4 to 5 years old and which had been on shorted storage for 3 to 4 years. These cells were tested to compare the response to the Gulton cells of the same vintage tested in Sequence 3.

After opening the circuit of these cells for the first time in several years, the voltage of the cells recovered to a range of 0.010 to 0.075V in 24 hours. Immediately thereafter, 3 minutes of charge at C/10 (without a prior full charge-discharge cycle) resulted in on-charge voltages ranging from 0.314 to 0.897 volt. After six minutes at C/10 the range (for Group 2 only) was 0.760-1.322 volts. When the circuit was opened, the voltages decayed rapidly. This response resembled that for the first open circuit stand of Test Sequence No. 2 shown in Figure 27, and was not plotted.

Following this, a voltage recovery test was done using an immediately preceding full charge and discharge, as prescribed in Reference 6, with the addition of a 4-hour dead shorted period following the 16 hour resistive discharge. The results on open circuit are shown in Figure 37. All voltages were above 1.1 volts within 3 hours after removing the shorts, and were above 1.21 volts at the end of 24 hours.

After another 4 hour dead shorted period, the cells were given a final voltage decay test (i.e., no charge-discharge cycle was performed after the voltage recovery test). The data is shown plotted in Figure 38. The trend line for the 3 minute charge is S-shaped, with a ledge around 12 hours, whereas that for the 6 minute charge is quite straight from 2 to 48 hours. The voltages of the two groups was essentially the same after 72 hours. Thus it appears that a voltage decay test may be

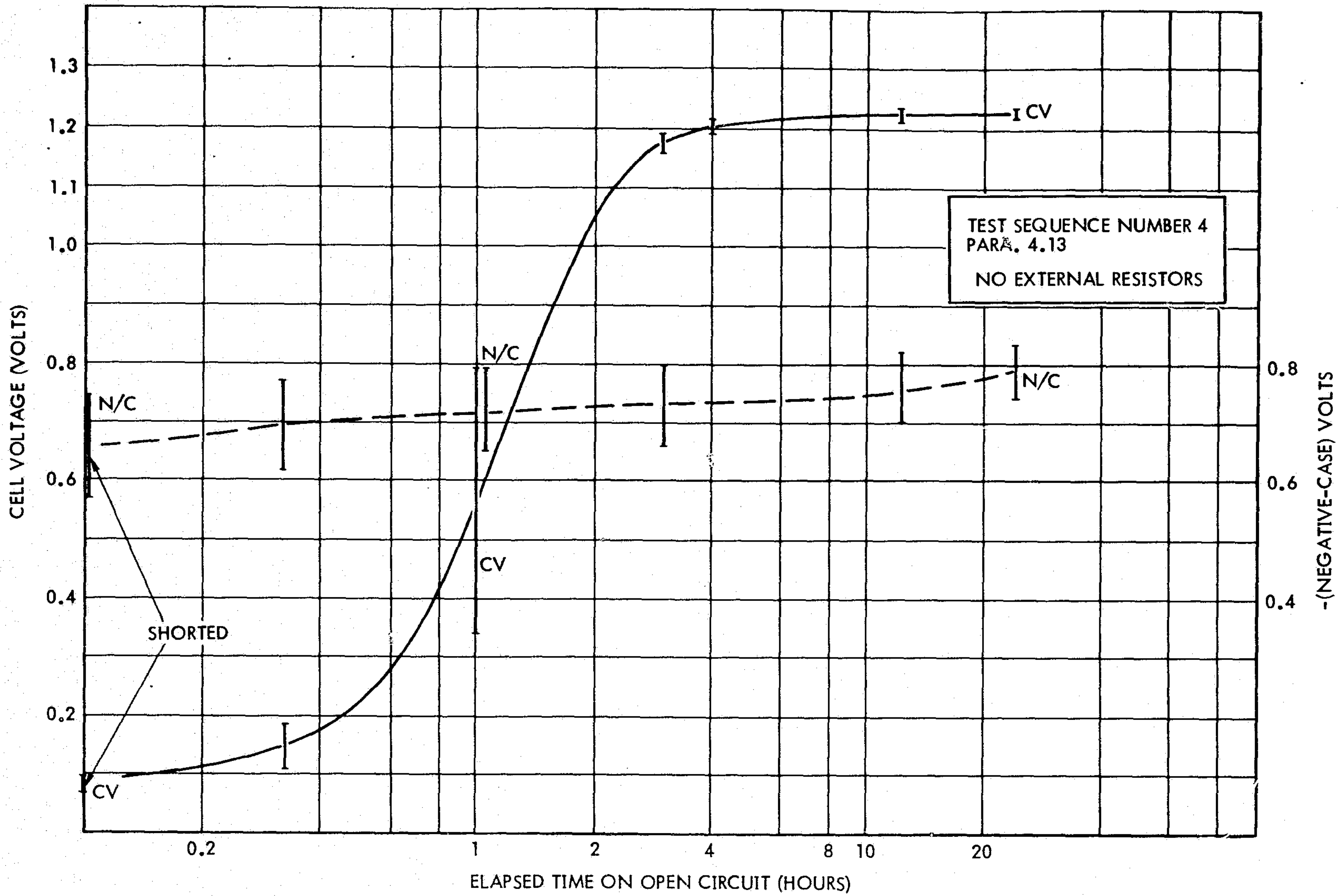

Figure 37. Voltage Recovery Plot, Test Sequence No. 4, Paragraph 4. 13

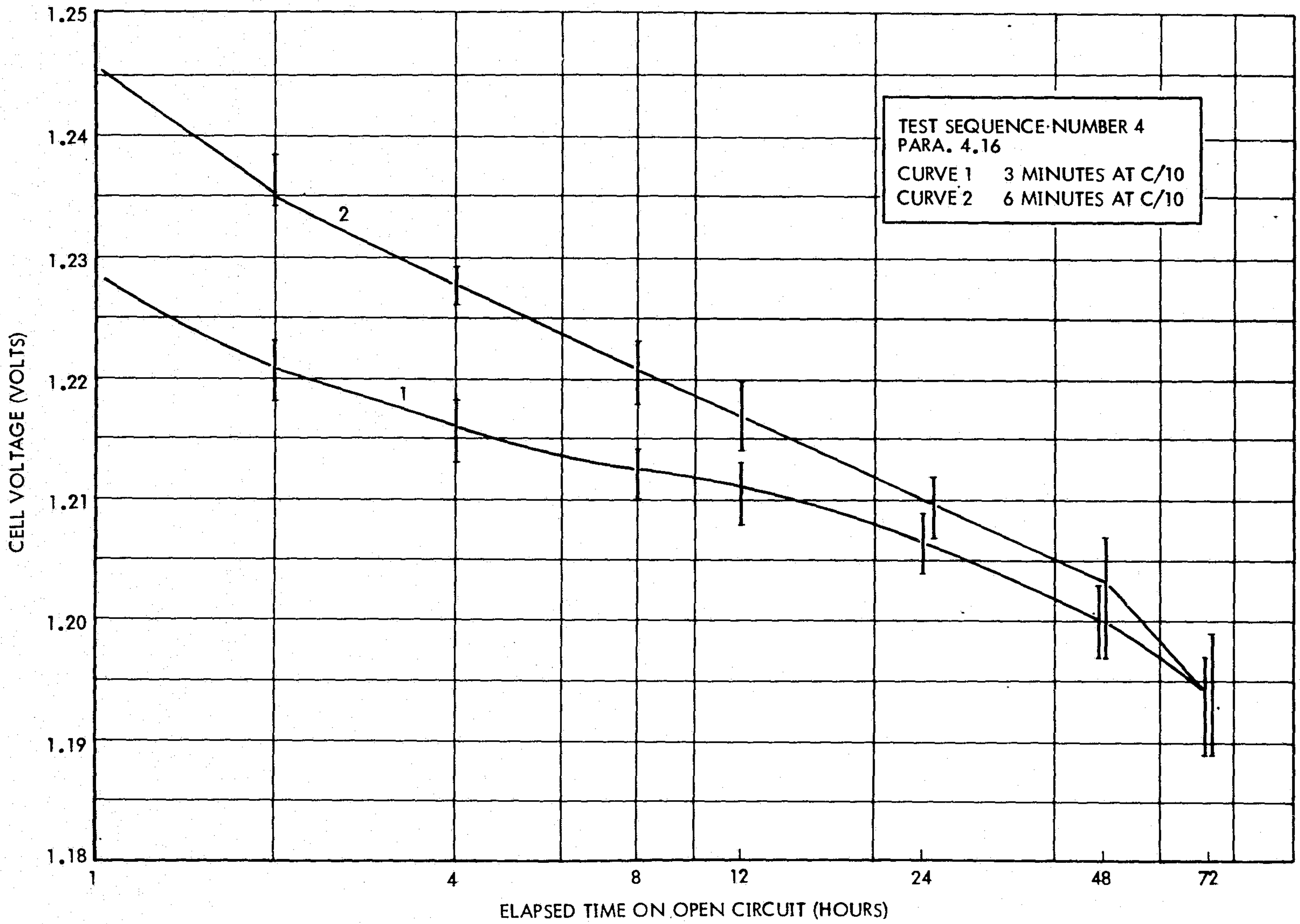

Figure 38. Voltage Decay Plot, Test Sequence No. 4, Paragraph 4.16

performed satisfactorily after a voltage recovery test, without an intervening cycle, provided the cells have been adequately conditioned prior to the voltage recovery test.

4.3.5 Results From Test Sequence No. 5

The general purpose of Test Sequence No. 5 is described in Paragraph 1 of this Test Procedure in the Appendix. All cells in this test lot contained built-in reference electrodes consisting of a small nickel oxide plate. Connection was made through a separate insulated terminal on the cover of the cell. Negative terminal to reference electrode terminal potential difference was measured in addition to cell voltage throughout this test sequence.

The cells were tested in "packs" of six. Each pack contained three cells with polypropylene and three with nylon separators. Packs were physically and electrically isolated from each other, but they were all bonded to the same heat sink. The cells in each pack were operated electrically in series and each pack was operated from a separate control circuit. As the test sequence for four of the packs (1 through 4) was different from that for the other four (5 through 8), the results for these two sets of packs are described separately.

4.3.5.1 Results From Pack No. 1 Through 4

At the end of 12 hours on 0.1 ohm resistors and four hours on dead shorts following the first charge of the test sequence (Procedure Para. 4.1(b)), the potential difference between the negative electrode and the reference electrode (abbreviated as Neg/Ref or (Neg-Ref)), ranged from -0.016 to -0.733 volts (with cells shorted).

Twenty minutes after removing the shorts, all cell voltages were near 1 volt. The voltage data for two of the four packs from hour 1 to hour 24 during the open circuit period are plotted in Figures 39 through 42. In these and subsequent figures cell voltage and the negative of the Neg/Ref voltage for individual cells are plotted together. The negative of the Neg/Ref voltage is used to facilitate visual comparison of the trends for the two voltages. Inasmuch as a negative-going negative electrode potential results in an increasing cell voltage, the plots as shown allow one to see easily the degree to which the cell voltage is a function of the negative electrode potential.

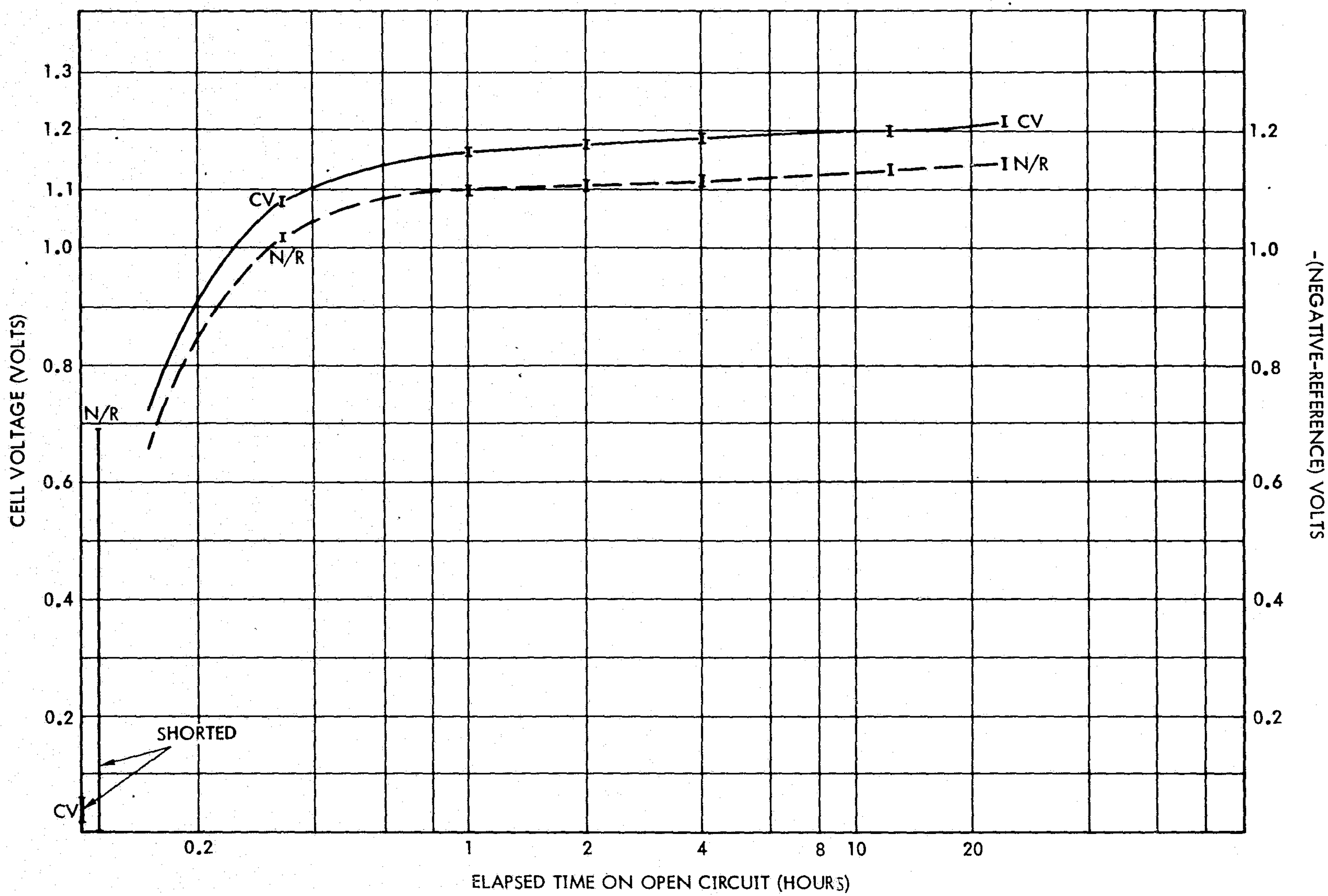

Figure 39. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(c)

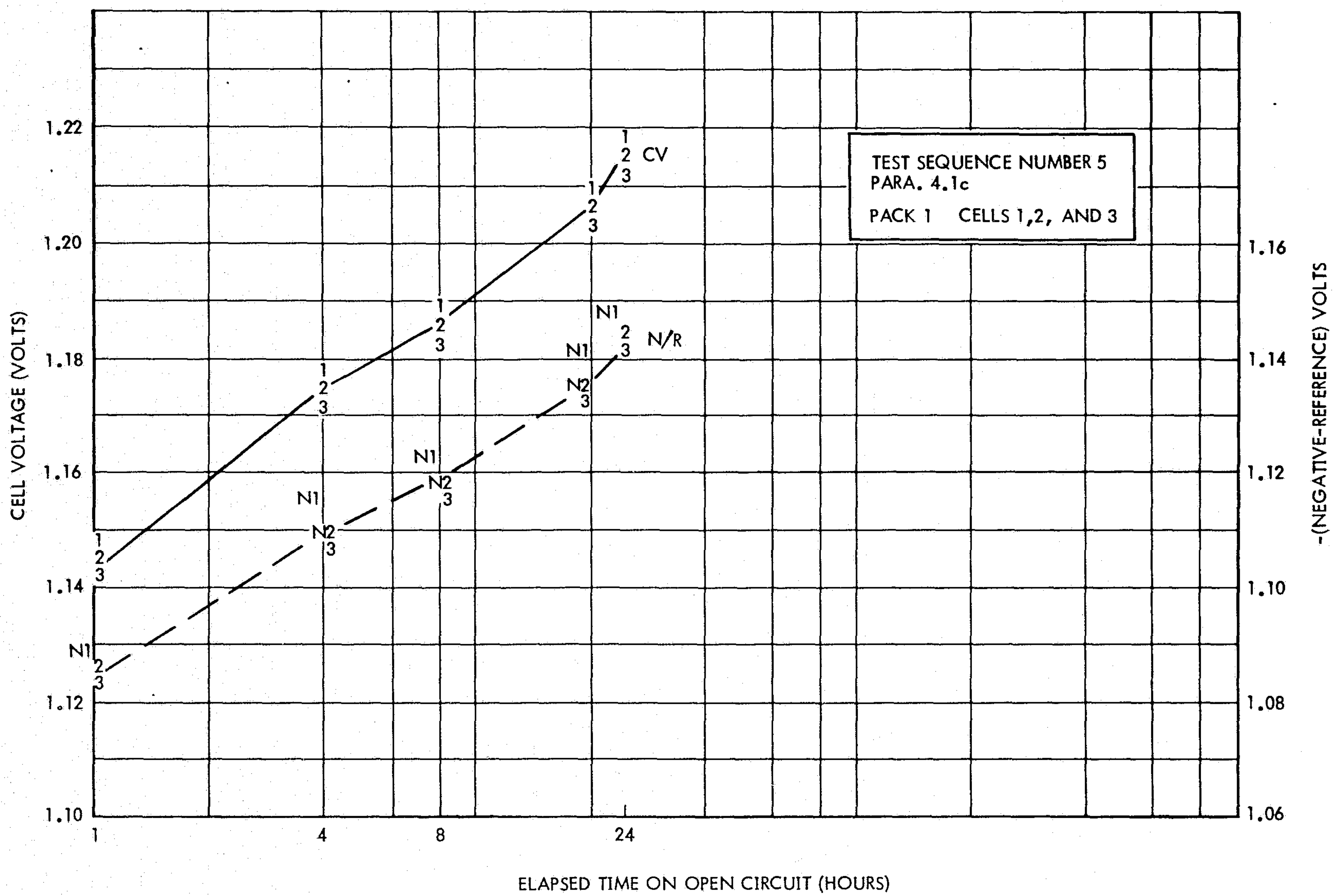

Figure 40. Expanded Plot, Test Sequence No. 5, Paragraph 4.1(c), Pack 1

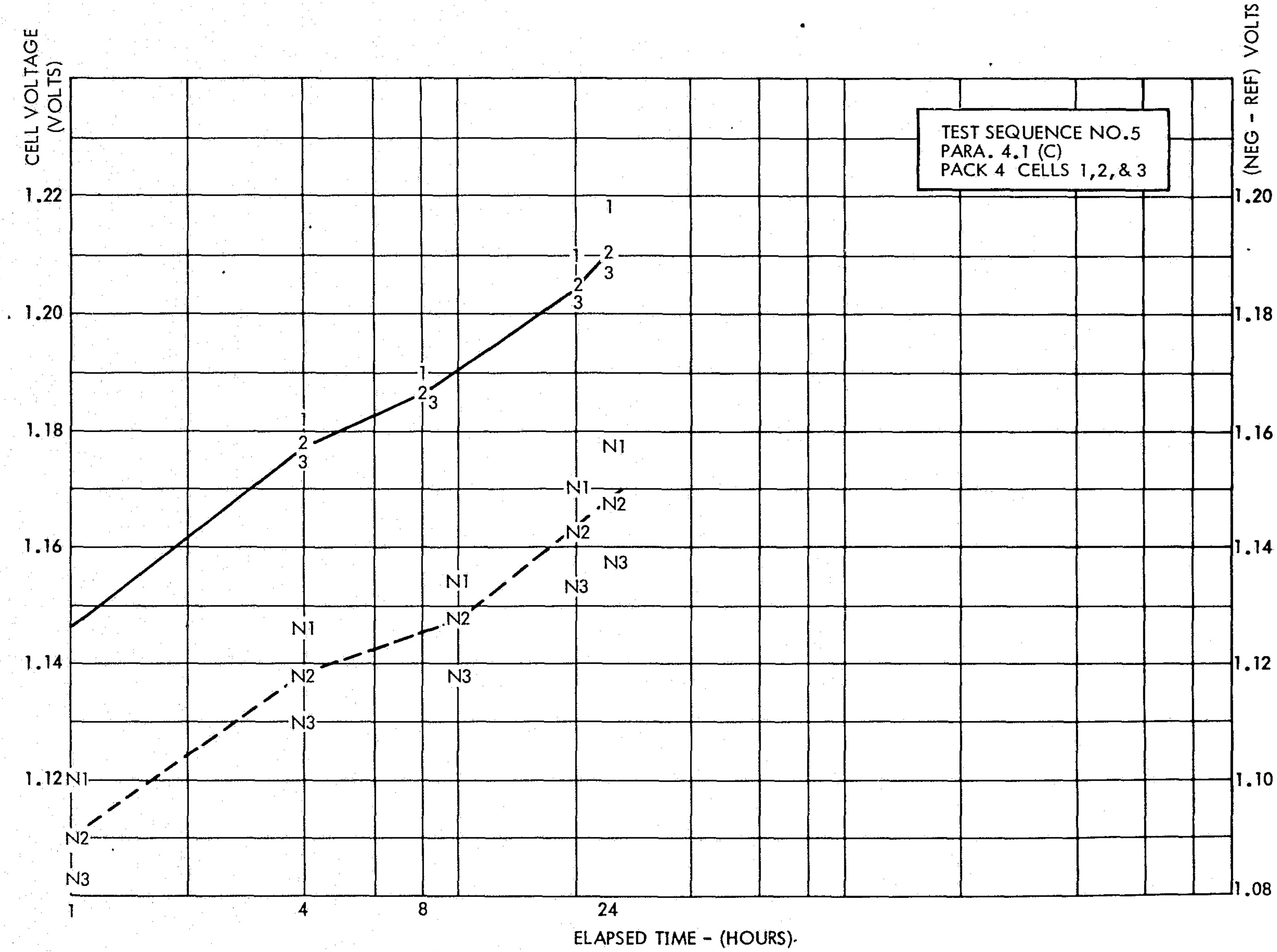

Figure 41. Expanded Plot, Test Sequence No. 5, Paragraph 4.1(c), Pack 4

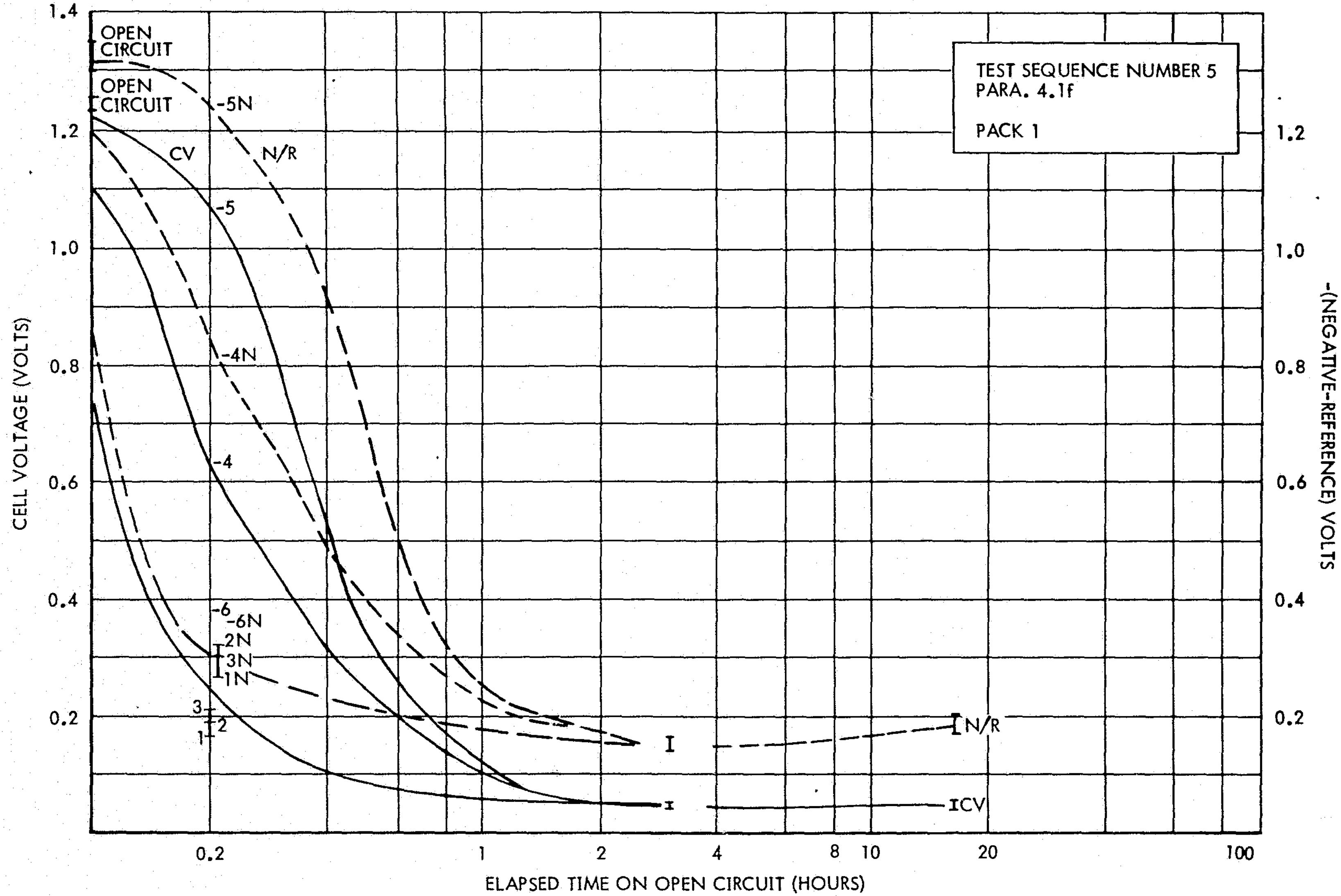

Figure 42. Voltage During Let-down, Test Sequence No. 5, Paragraph 4.1(b)

In these figures and those to follow, Neg/Ref voltage data are often plotted on a different scale than are the cell voltages. The scale used has the same scale factor (volts per unit distance on the chart) but is displaced with respect to the cell voltage scale as necessary to clarify the presentation.

It may be seen tha tthe actual values of Neg/Ref voltage at any point on the time scale vary considerably from cell to cell. This is due to the fact that the different reference electrodes are at different half-cell potentials because they are at different states of charge. Despite these differences, the half-cell potential of the isolated nickel oxide electrode in any one cell was believed to be constant within a few millivolts over the time required for the various open circuit tests, and hence the changes shown in the Neg/Ref voltage during any run are believed to be accurate and valid.

Note in Figures 39-41 that the shapes of the curves are quite different from those obtained during Test Sequences 3 and 4, and that the cell voltages were still increasing at the end of 24 hours on open circuit. Although the semilog plot makes the slope at the end appear steeper (relative to that earlier in the test) than would be the case if a linear time scale were used, the slopes in volts per hour are in fact greater for the last eight hours than for the previous eight hours. Also, the cell voltage curves are close to being parallel with the Neg/Reg voltage curves, indicating that the negative electrodes in these cells were largely responsible for the cell voltage behavior.

After the next full charge-discharge cycle, 0.1 ohm resistors were applied for 16 hours. (Procedure Para. 4.1(f)). Cell voltages and Neg/Ref voltages during this time are plotted in Figure 43 for one representative cell pack. It may be seen that cell voltages all had gone below 0.1 volt in less than 2 hours after connecting 0.1 ohm resistors, and remained essentially constant from the second hour until the end of the 16 hour stand. The Neg/Reg voltage curves closely followed the cell voltage curves, with a slight increase toward the end of the stand period.

After the 0..1 ohm resistors were removed (resistors of 1000 ohm, 500 ohm and 100 ohms then remained on packs 2, 3 and 4 respectively), all cell voltages rose to over 0.7 volt in ten minutes. The data for the

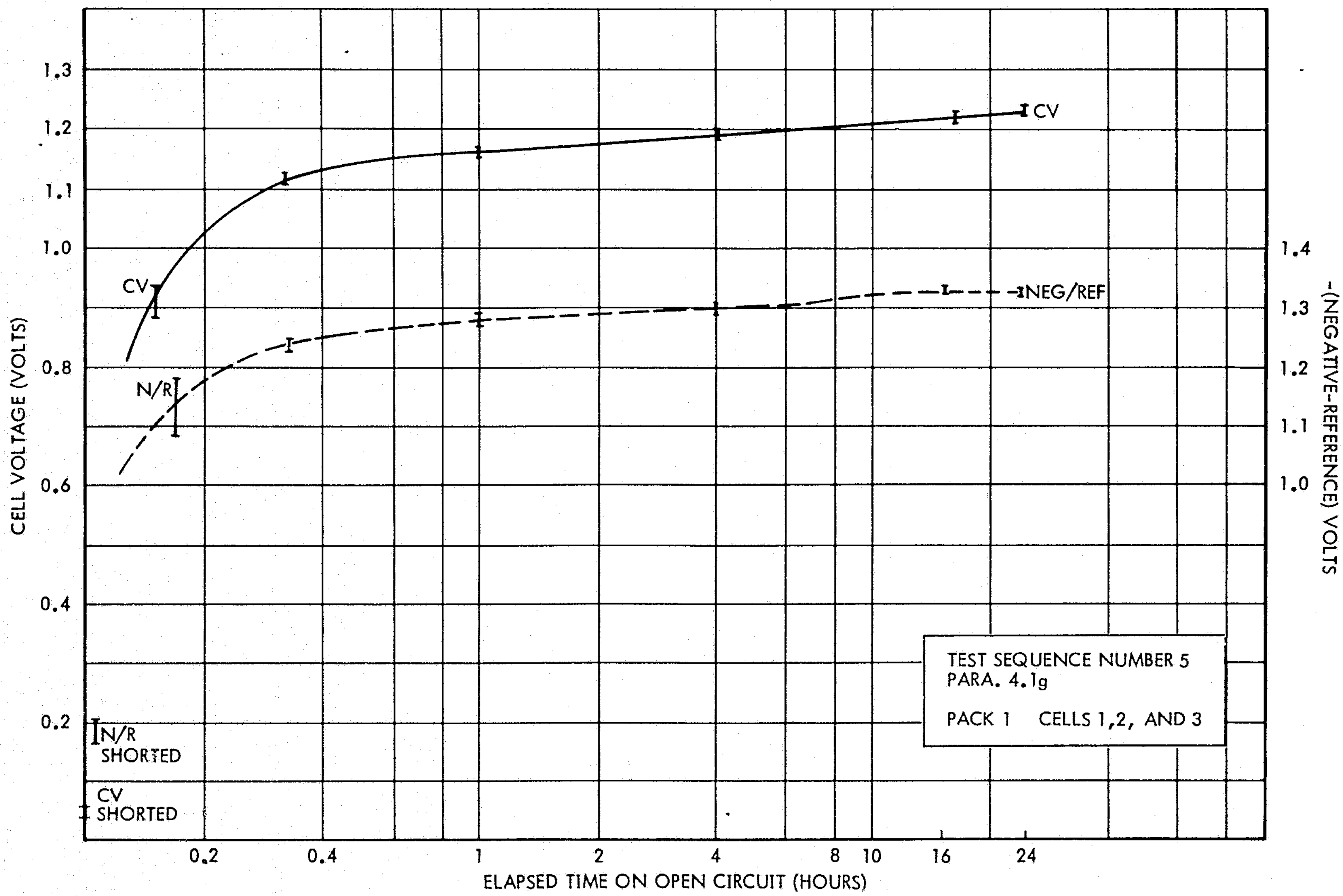

Figure 43. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(g), Pack 1

cells having polypropylene separators in pack 1 (no resistors present) and pack 4 (100 ohms attached) are shown plotted in Figures 43 through 46. Data for the Pack 4 cells having nylon separators are shown in Figure 47. Note here that the voltages for Pack 1 were generally higher than those during the first test described above.

After the above open-circuit stand, the cells were shorted for 4 hours. At the end of this time, with the shorts on, the following Neg/Ref voltages were observed:

Range of Neg/Ref Voltage (V)

Pack No.	Cells Nos. 1, 2, and 3	Cells Nos. 4, 5, and 6
1	-0.259 to -0.566	-0.593 to -1.239
2	-0.156 to -0.303	-0.594 to -1.305
3	-0.215 to -0.545	-0.179 to -1.287
4	-0.192 to -0.417	-0.650 to -1.308

Note the greater negative values and wider range for cell 4, 5 and 6. At the end of the following 6 minute charge at 5A (Para. 4.1(i)) the following on-charge cell voltage data was recorded:

		Cells 1-3	Cells 4-6
Packs	1, 2 and 3	1.333 - 1.343	1.345 - 1.357
Pack	4	1.325 - 1.341	1.346 - 1.368

Voltages during the following open circuit stand period are shown plotted for 100 hours in Figures 48 and 49. Here again the shapes of these curves are very different from those shown for the smaller cells. Note that for Pack 1 (no resistors added) the voltages decreased at first, then increased until about 48 hours, after which the voltages of cells 1, 2 and 3 continued to increase while those of cells 4, 5 and 6 began to decrease. Here again cell voltage changes paralleled those of the negative electrodes for the first 48 hours, then began to diverge. Note also that the cell voltages of cells 5 and 6 started higher but ended lower than the rest.

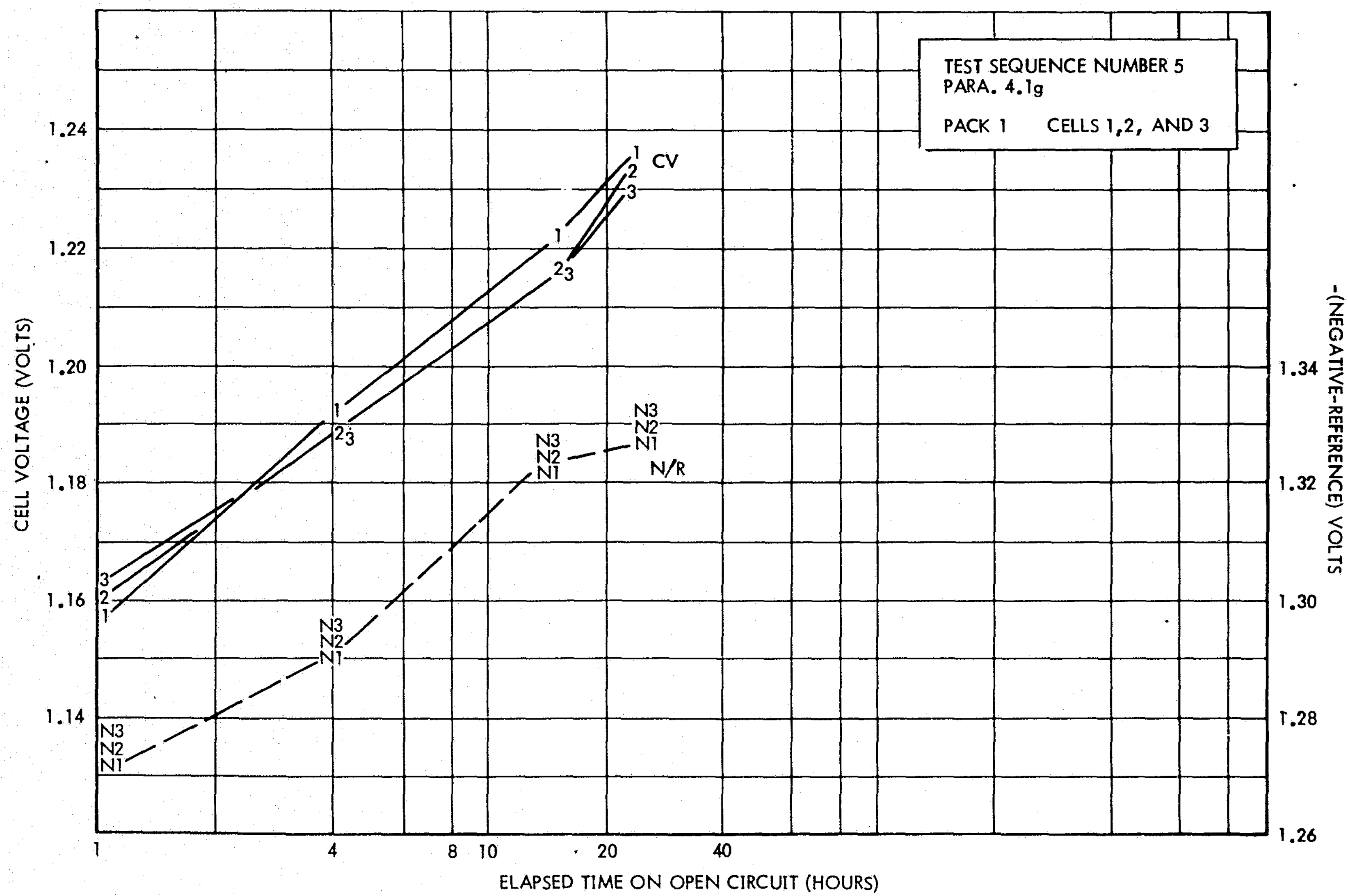

Figure 44. Expanded Plot, Test Sequence No. 5, Paragraph 4.1(g), Pack 1

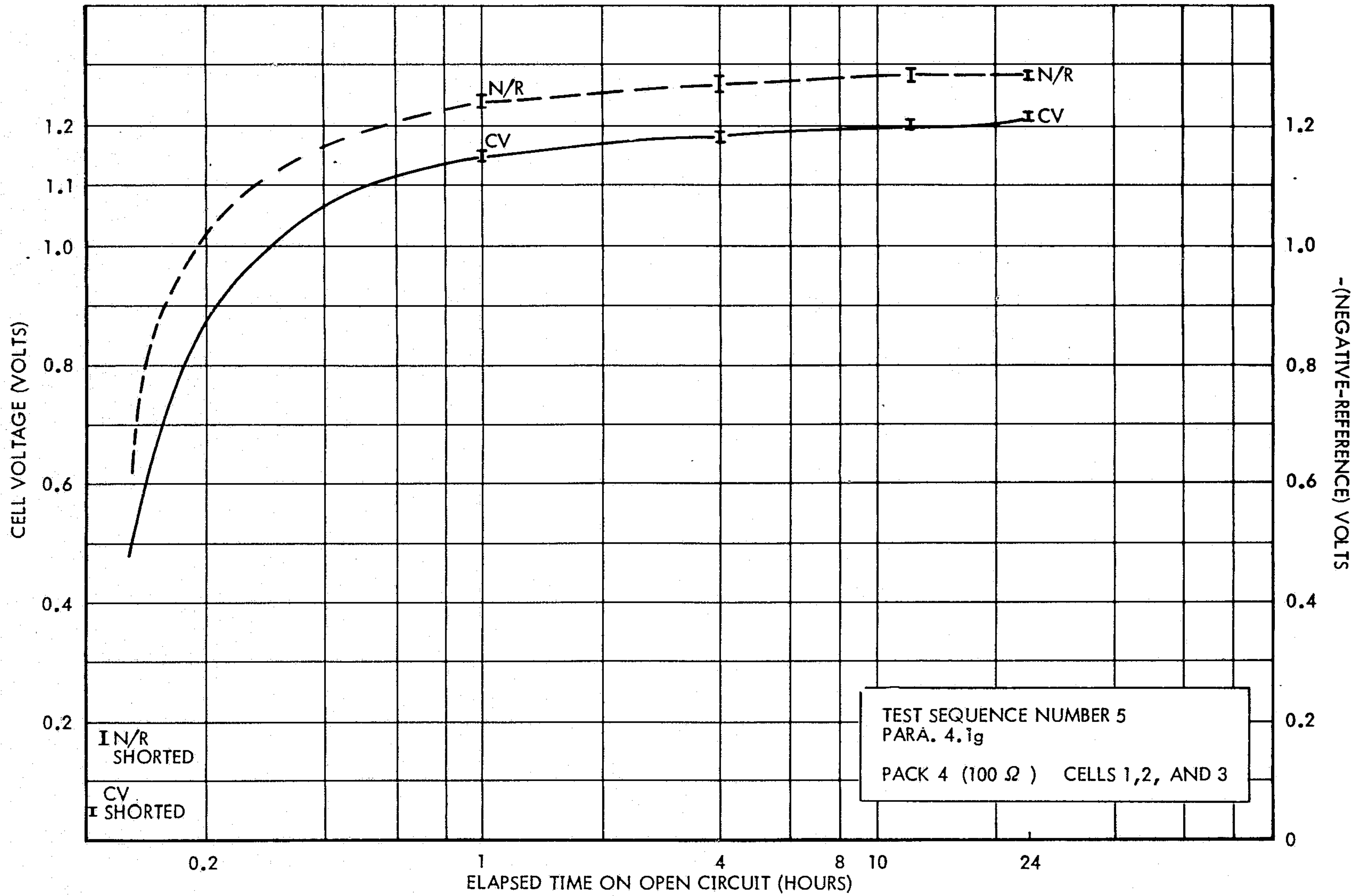

Figure 45. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(g), Pack 4

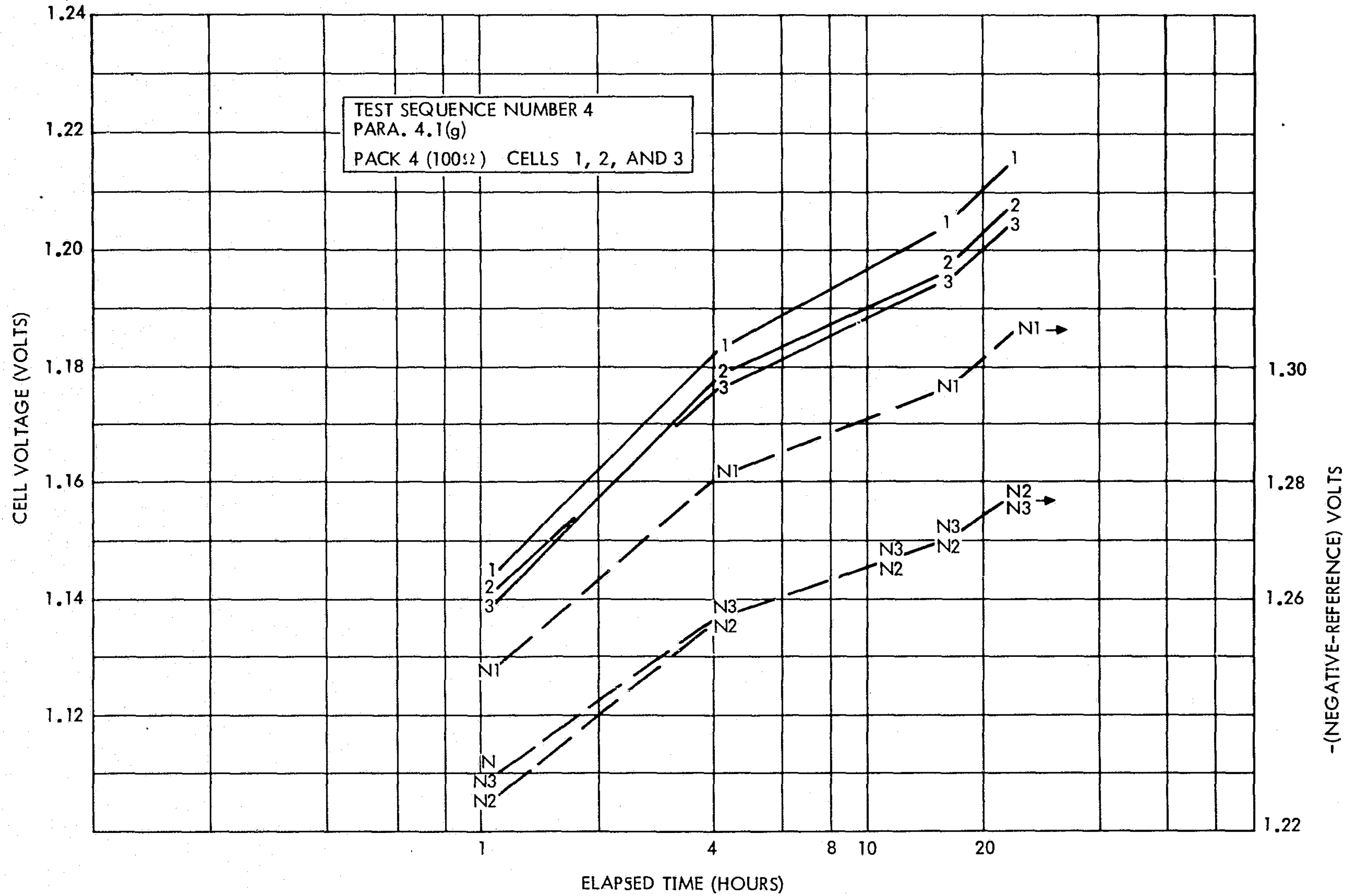

Figure 46. Expanded Plot Test Sequence No. 5, Paragraph 4.1(g), Pack 4 Polypropylene Separators

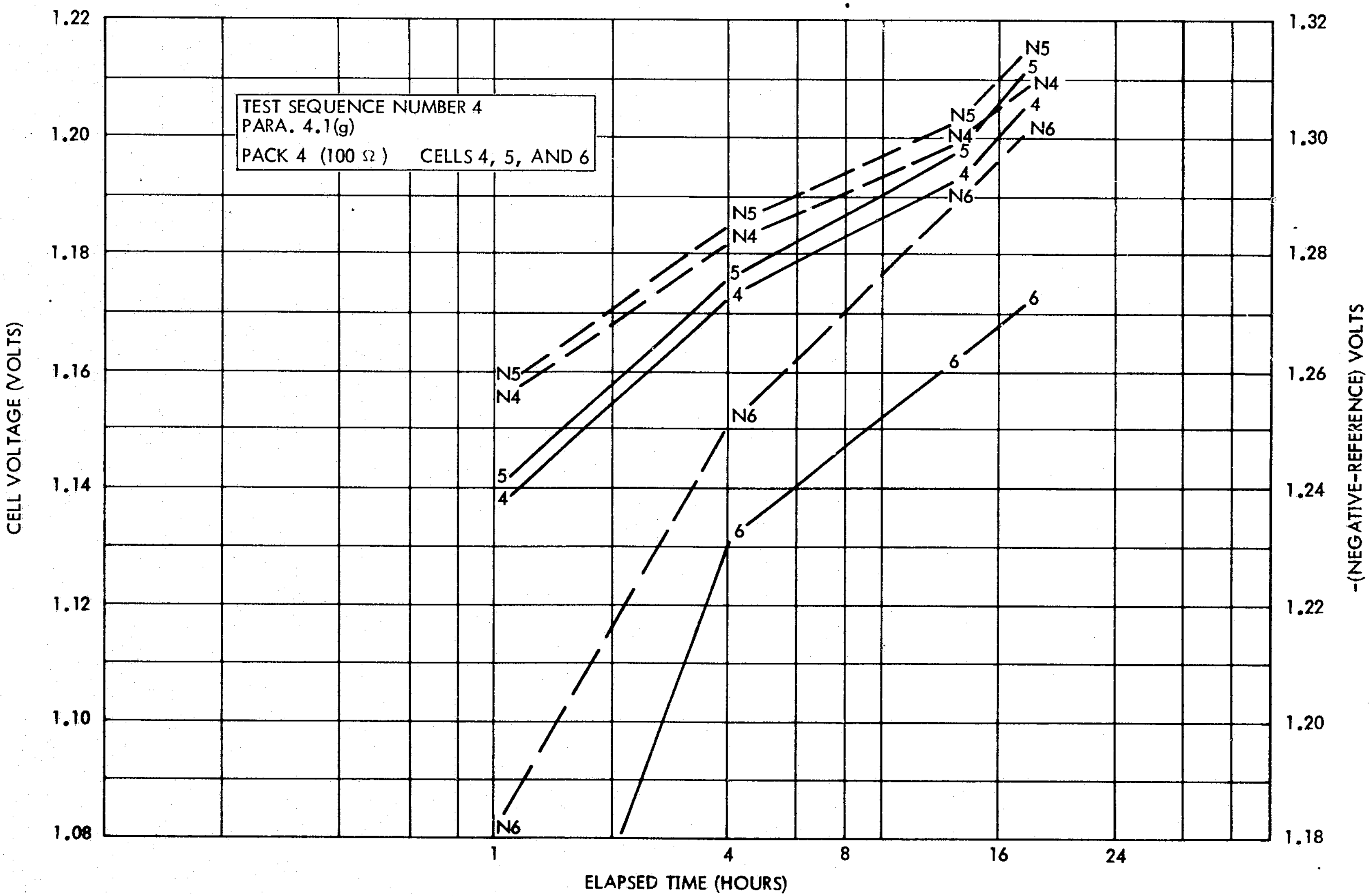

Figure 47. Expanded Plot, Test Sequence No. 5, Paragraph 4.1(g), Pack 4 Nylon Separators

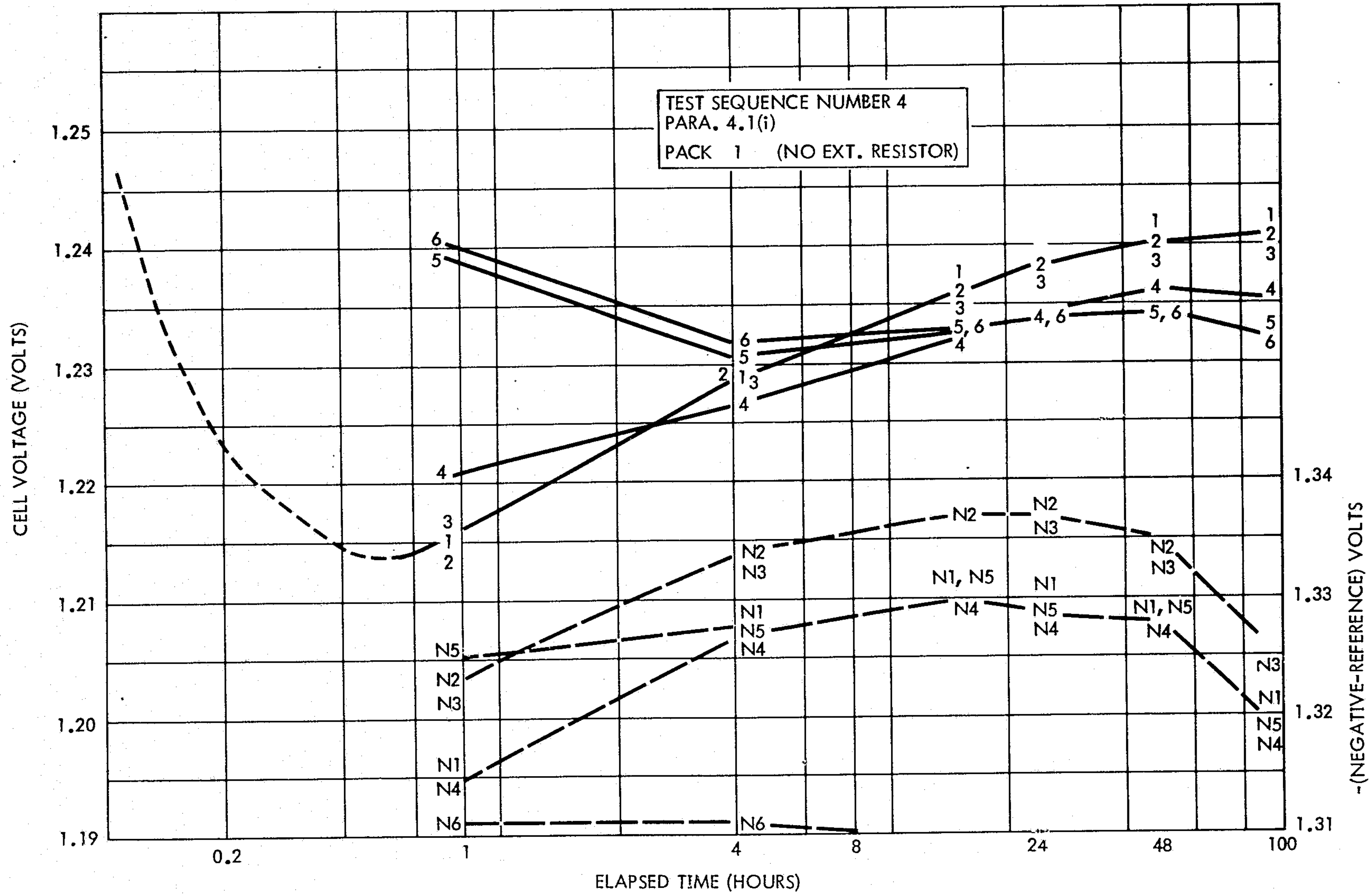

Figure 48. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(i), Pack 1

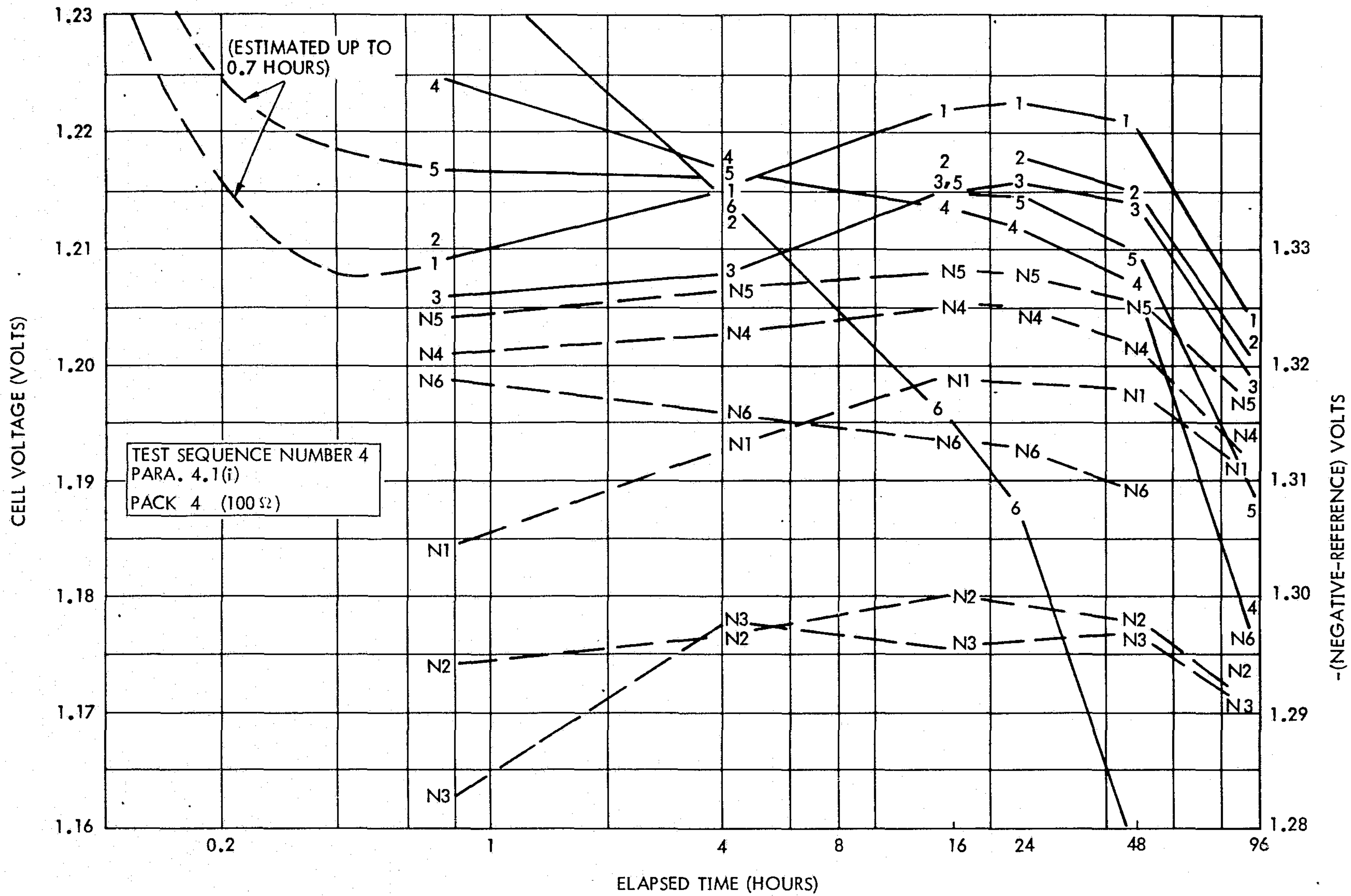

Figure 49. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(i). Pack 4

In Figure 49, (Pack 4, 100 ohms attached) the curve for cell 1 appears similar to that in Figure 48 up to 24 hours, but the rest of the curves are lower in voltage, flatter, and all begin to drop off after 24 hours on open circuit. Cell 6 is obviously an extreme case, starting very high and ending very low. It is interesting that even with this steep slope the cell voltage at 24 hours was as high as 1.188 volts.

At this point the value of the resistance used for the low-resistance discharge was changed from 0.1 ohm to 0.25 ohm to obtain comparative data for a different resistance. After a few hours on 0.25 ohm resistors following the above open circuit stand, Neg/Ref voltages were all more negative than -1.28 volts, where they remained throughout a stand of four days. At that point cell voltages ranged from 0.035 to 0.075 volts.

During the let-down period, following the next full charge and discharge (Procedure Para. 4.1(k)), resistors of 100, 50, 20 and 250 ohms were installed on Packs 1, 2, 3 and 4 respectively. There lower resistors were tried in a continued attempt to span the usable sensitivity of these tests in view of the apparent insensitivity of the tests performed earlier with resistances of 1000, 500 and 100 ohms.

A plot of voltage data for cells 1, 2 and 3 of Packs 1 and 4 during 24 hours on 0.25 ohms is shown in Figure 50. Here again the cell voltages closely parallel the Neg/Ref voltages (with the sign changed) for the first 12 hours, after which the negative potentials began to increase as the cell voltages continued to decrease.

Packs 1-4 were then subjected to a 6 minute C/10 charge and placed on open circuit for 48 hours. Plots of the voltages for packs 1, 2 and 4 are shown in Figures 51, 52 and 53. In some respects these curves resemble those from the Voltage Recovery Tests shown in Figures 46 and 47, in that voltages were increasing for most of the first 24 hours.

The curves for cells in pack 1 (100 ohms attached) are similar in shape to those for the cells with 100 ohms attached during the previous voltage decay test (Figure 48), but the cell voltages at 24 hours (five highest voltage cells) were 10-15 mV lower this time. The curves for four out of six cells in pack 2 (50 ohms) were slightly lower than those for 100 ohms, but probably would be hard to separate from 100 ohm responses

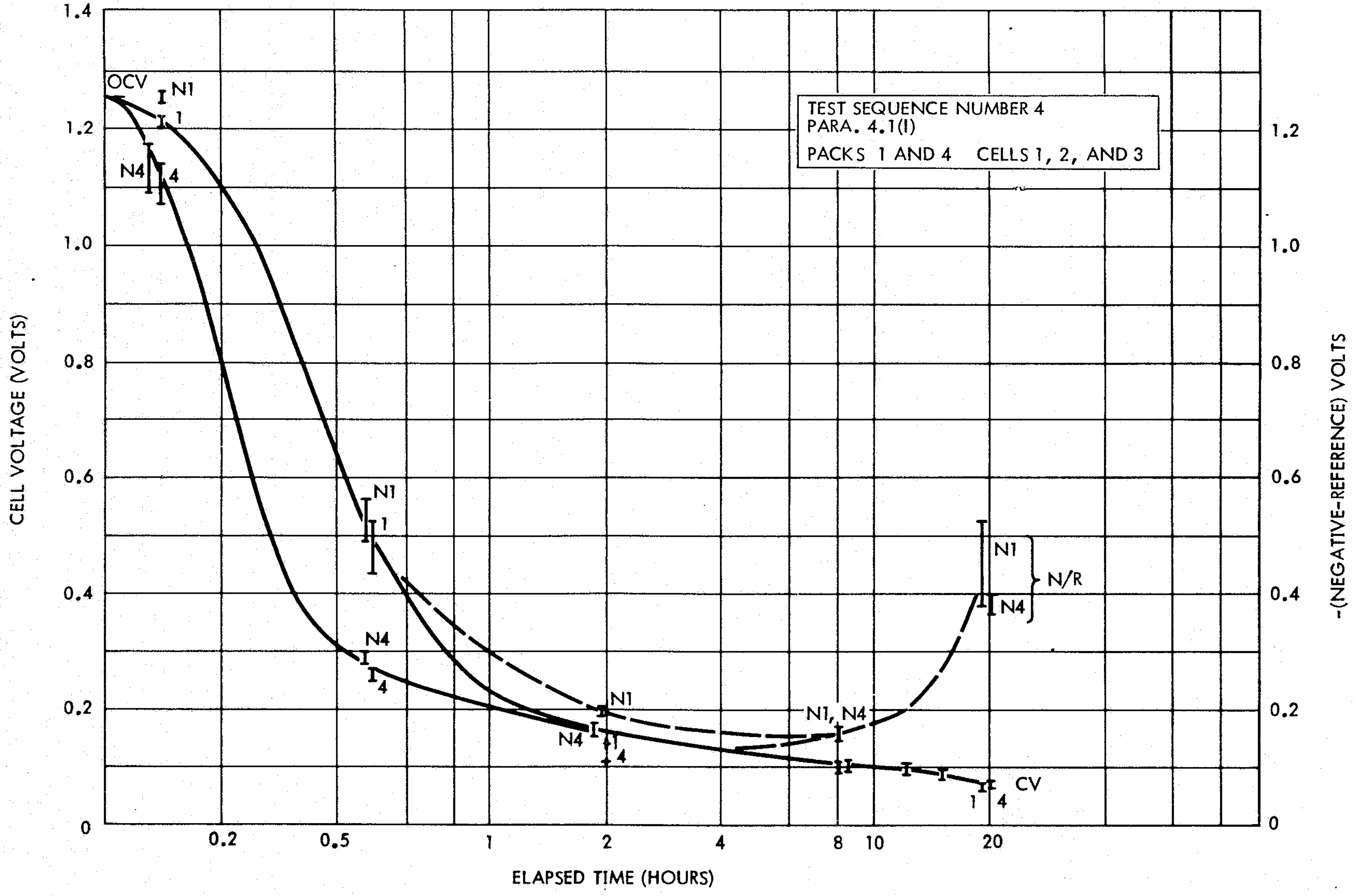

Figure 50. Voltage Data During Let-down, Test Sequence No. 5, Paragraph 4.1(1)

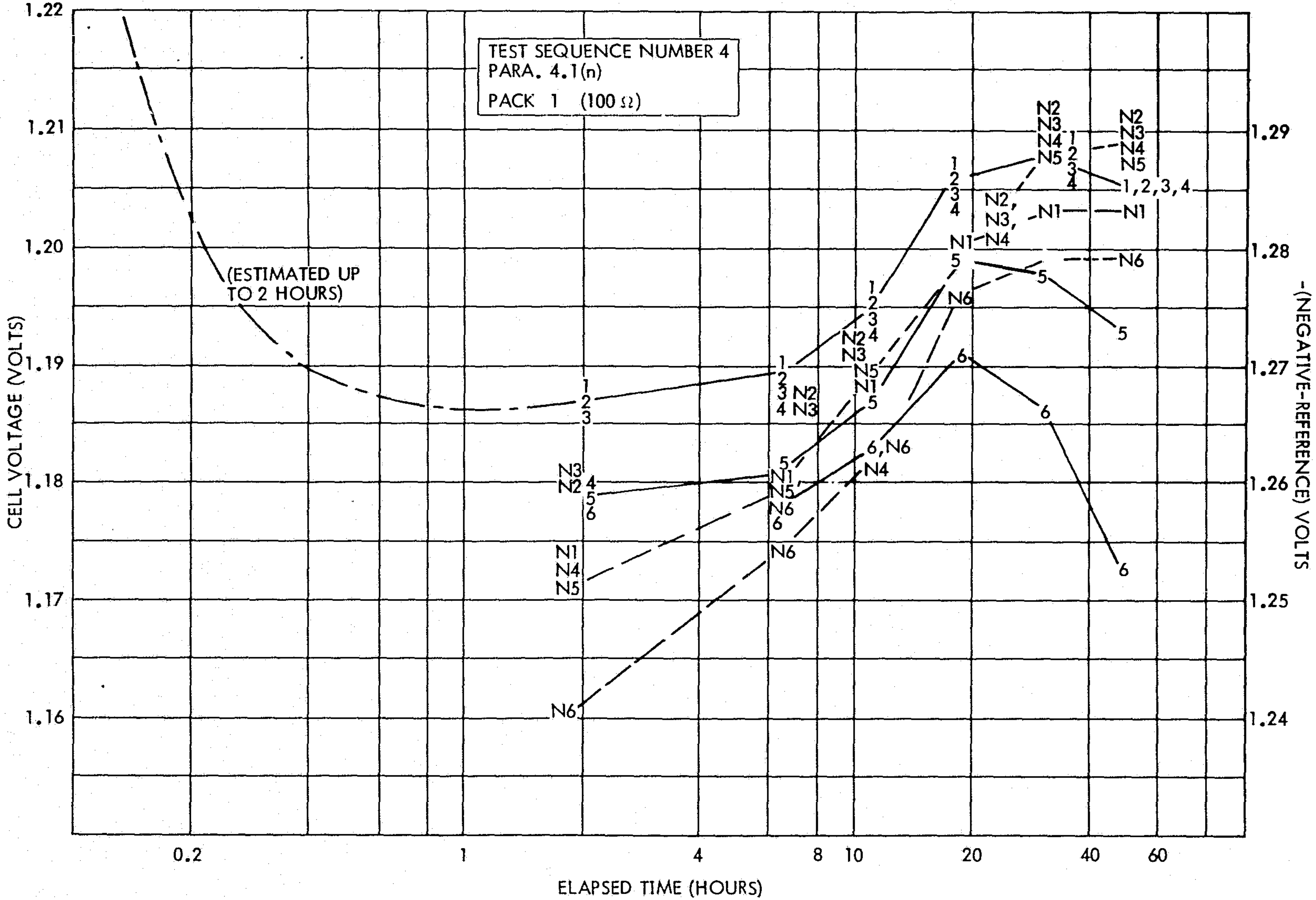

Figure 51. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(m), Pack 1

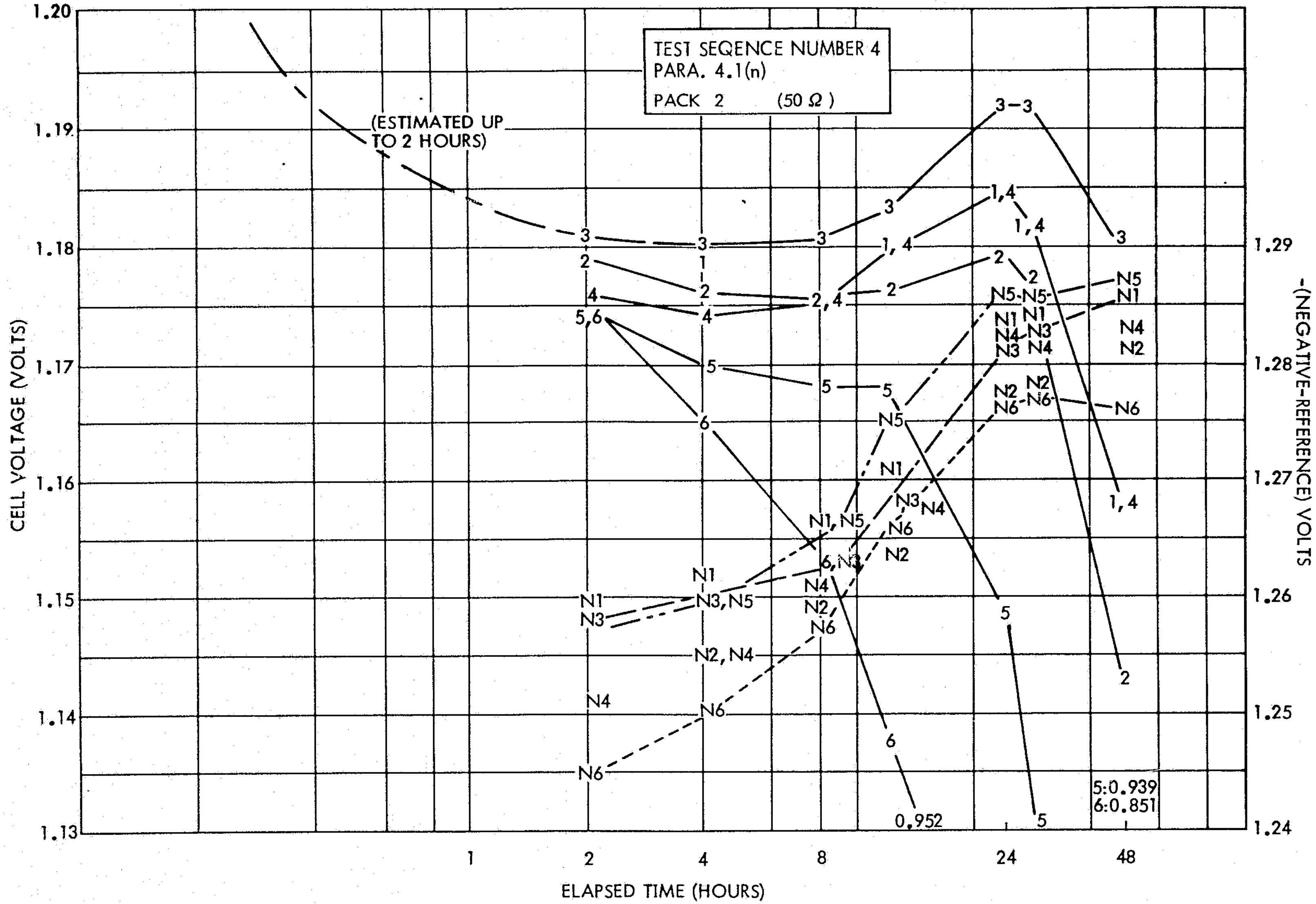

Figure 52. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(n), Pack 2

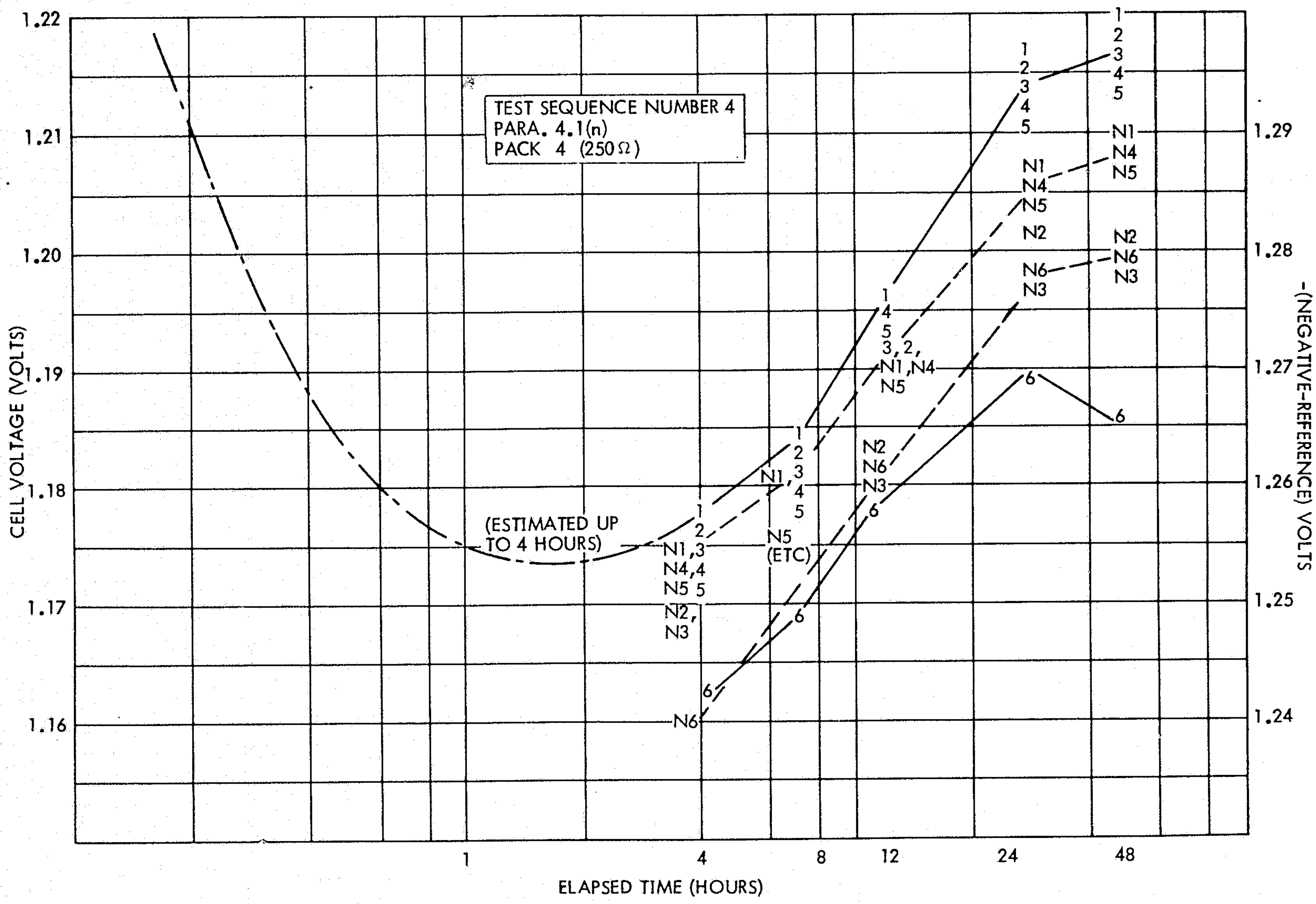

Figure 53. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(n), Pack 4

in practice. On the other hand, the voltages of five out of six cells in pack 4 (250Ω) were still increasing at 48 hours, although starting to level off.

In view of the fact that relatively large changes in the potential of the negative electrode were still seen to be occuring during these open circuit stand periods, resistors (0.25 ohm) were attached to these cells directly after the above test and left for 72 hours, in order to try to stabilize the negative electrode potential. At the end of this time cell voltages ranged from 0.002 to 0.004 volt, and Neg/Reg voltages from -1.27 to -1.28 volts.

When the 0.25 ohm resistors were removed for the next open circuit test, there was no immediate change in the Neg/Ref voltages.

Figures 54 and 55 show the voltage responses on open circuit. Note in Figure 54 that the shape of these voltage-time curves are similar to those of the smaller cells. Cell voltages increased much more slowly during the first 10 hours than in previous tests, on these cells, and were constant for the last eight hours. Neg/Ref voltages did not change appreciably, also in contrast to previous tests. Again the voltages of the nylon cells (Nos. 4, 5 and 6 in each pack, Figure 55), were widely scattered, and two of the total of nine cells shown in Figure 55 were below 0.9 volt after 24 hours.

After this, the test cells were subjected to a full charge-discharge cycle and then put on 0.25 ohm resistors for 48 hours. This amount of time is three times that called for in presently used procedures, and was provided to attempt to again stabilize the negative electrode potential after the recharge and discharge. The results are typified by the data shown in Figure 56 for packs 1 and 4. The voltage response was about the same as that shown in Figure 50 over the first 24 hours. After 24 hours the cell voltages (and hence currents) decreased by a factor of 20 while the Neg/Ref voltages changed by more than 1 volt, to reach a value of -1.25 volts, after a total stand time of 40 hours.

Following this, packs 1 and 2 were charged for 6 minutes at 5A and put on open circuit. Figures 57 and 58 show the open circuit voltage data for pack 1 (no resistors) and pack 2 (250 ohms added) respectively. Note,

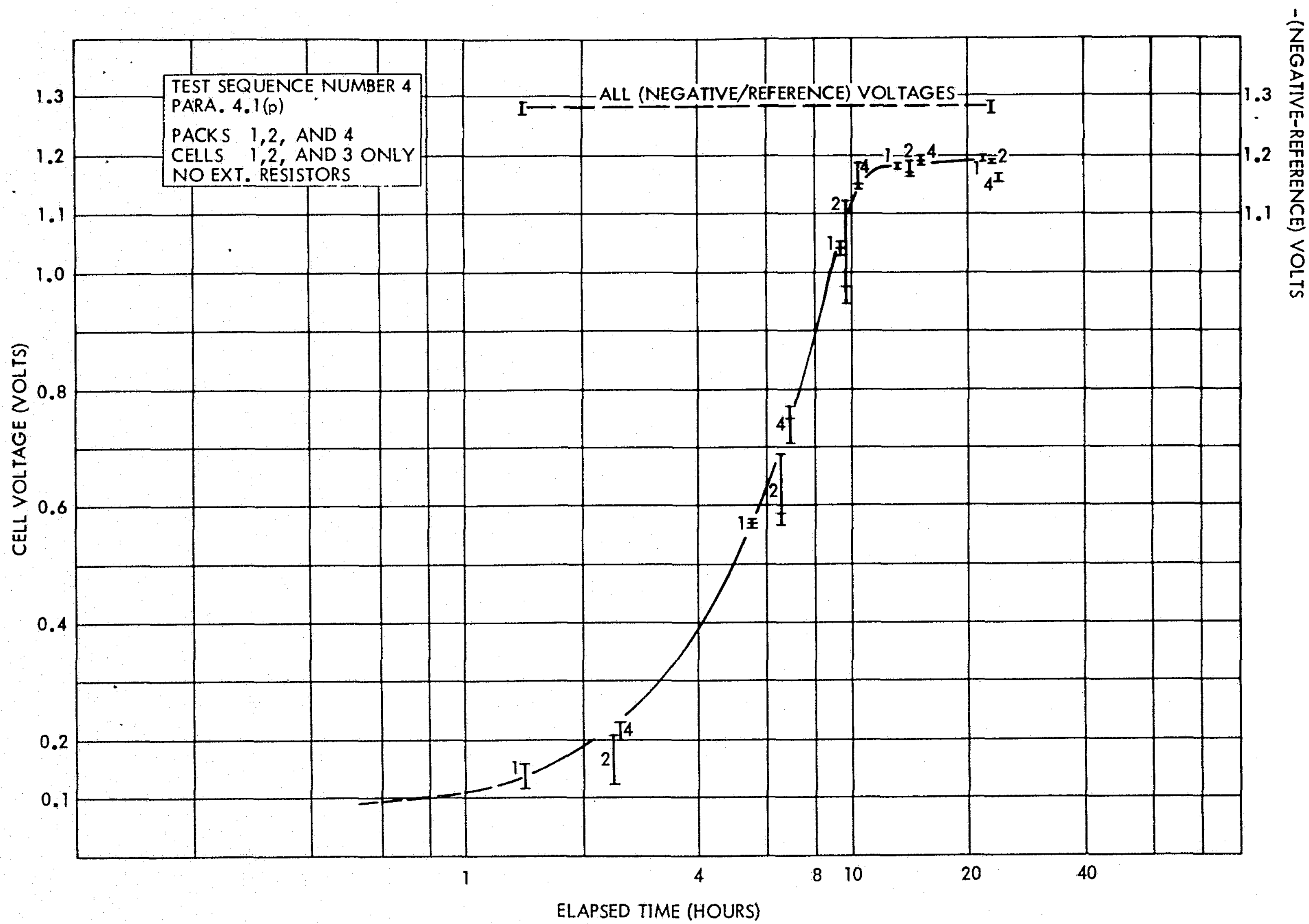

Figure 54. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(p), Polypropylene Separators

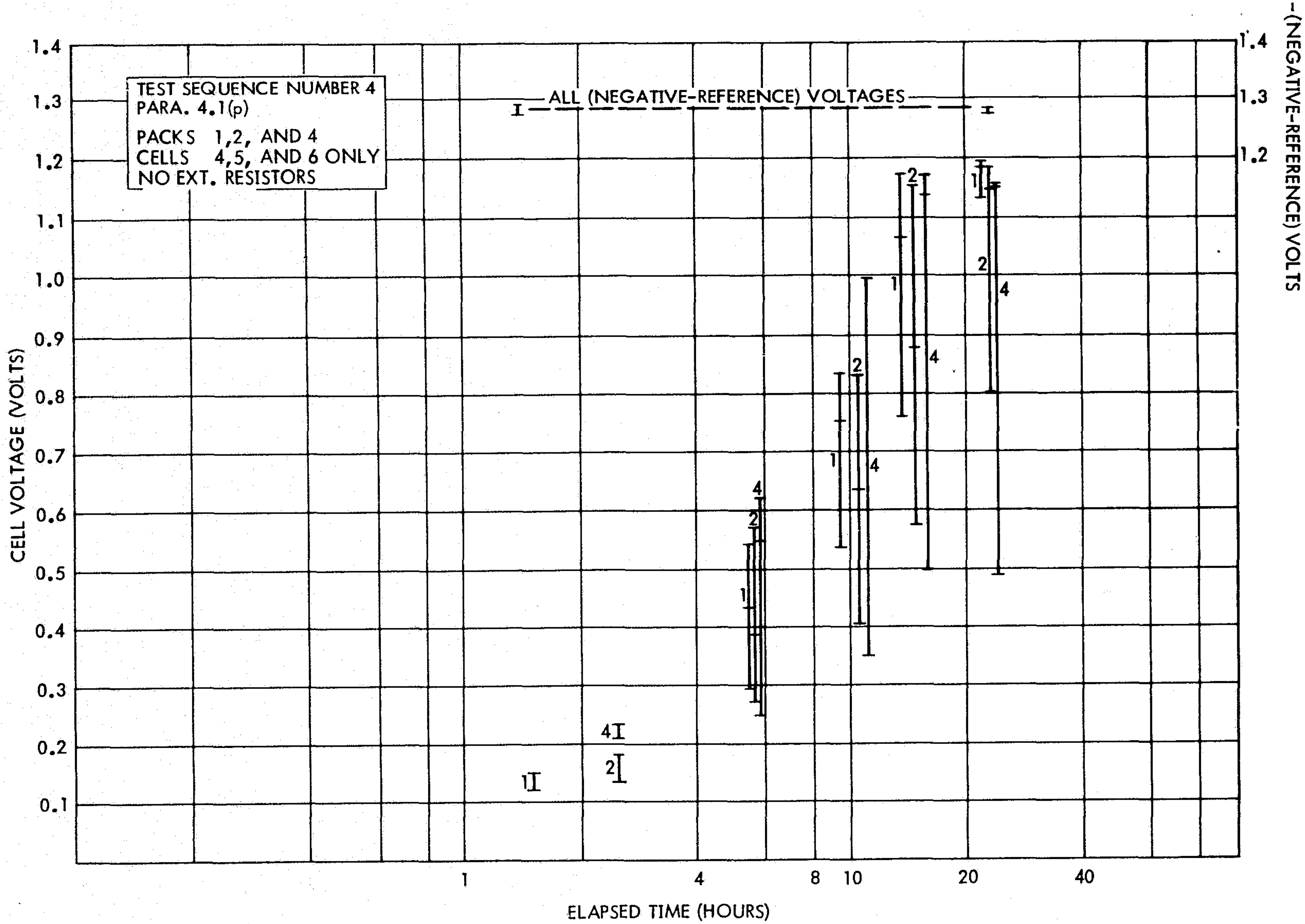

Figure 55. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(p), Nylon Separator

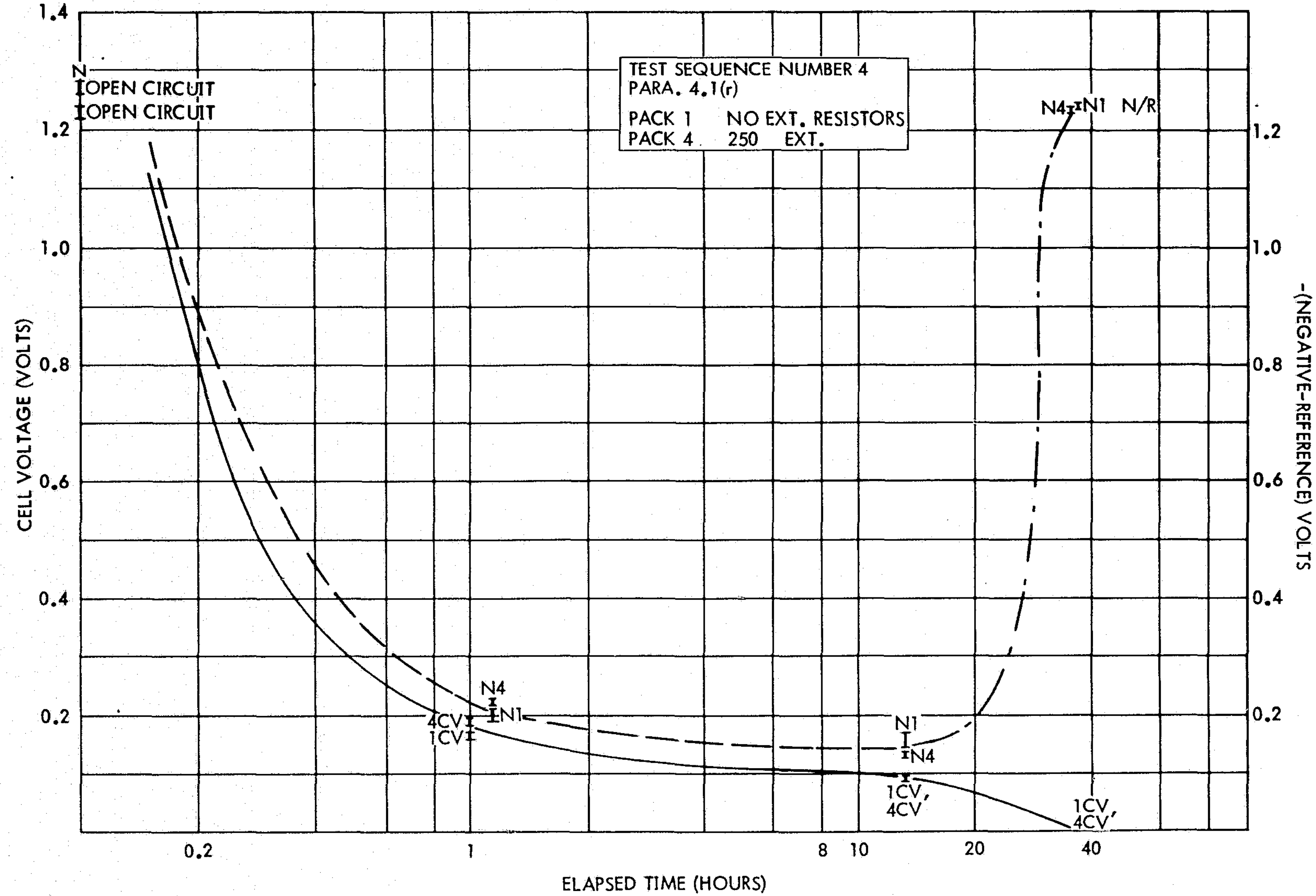

Figure 56. Voltage Response During Let-down, Test Sequence No. 5, Paragraph 4.1(r)

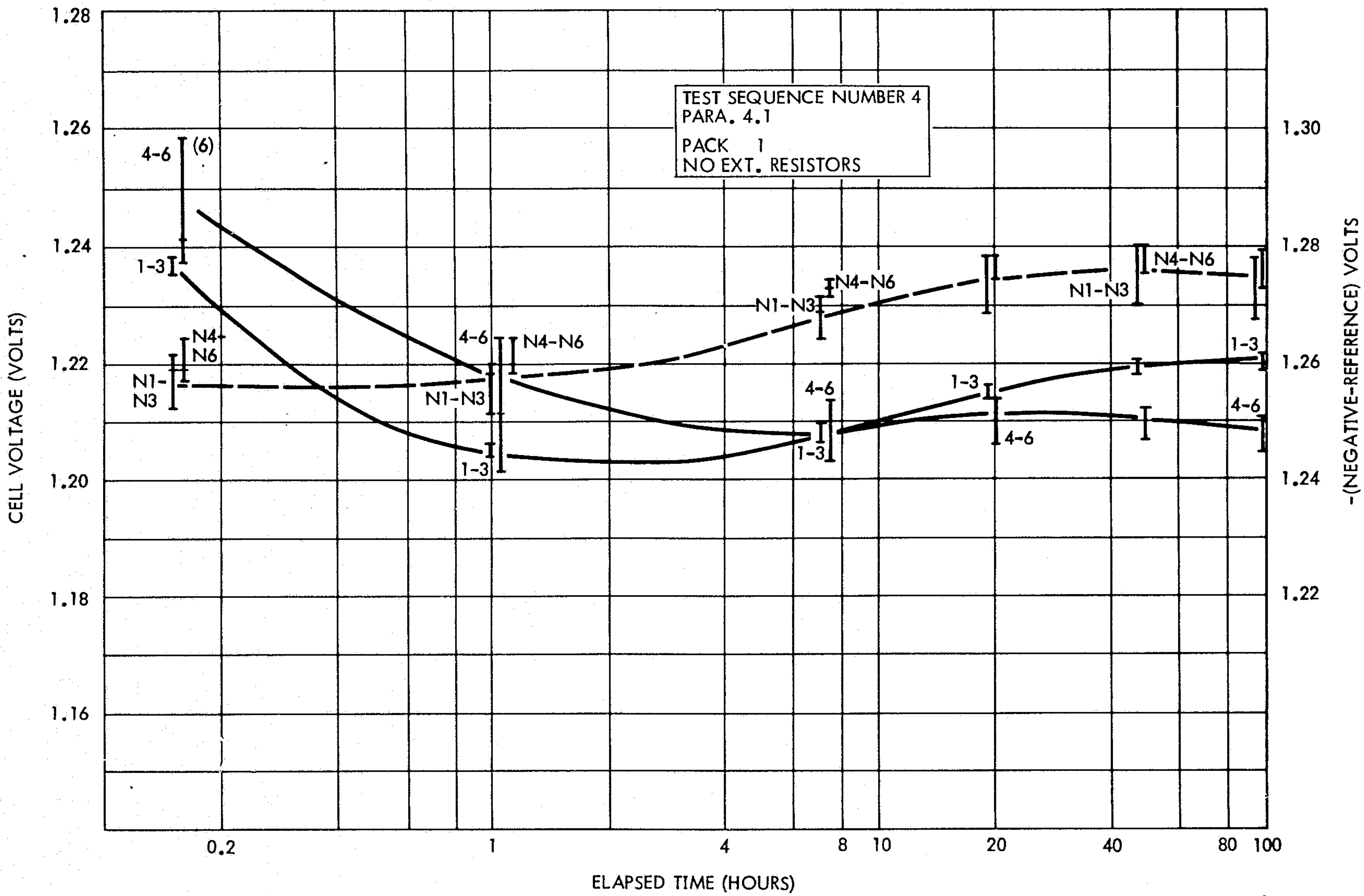

Figure 57. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(s), Pack 2

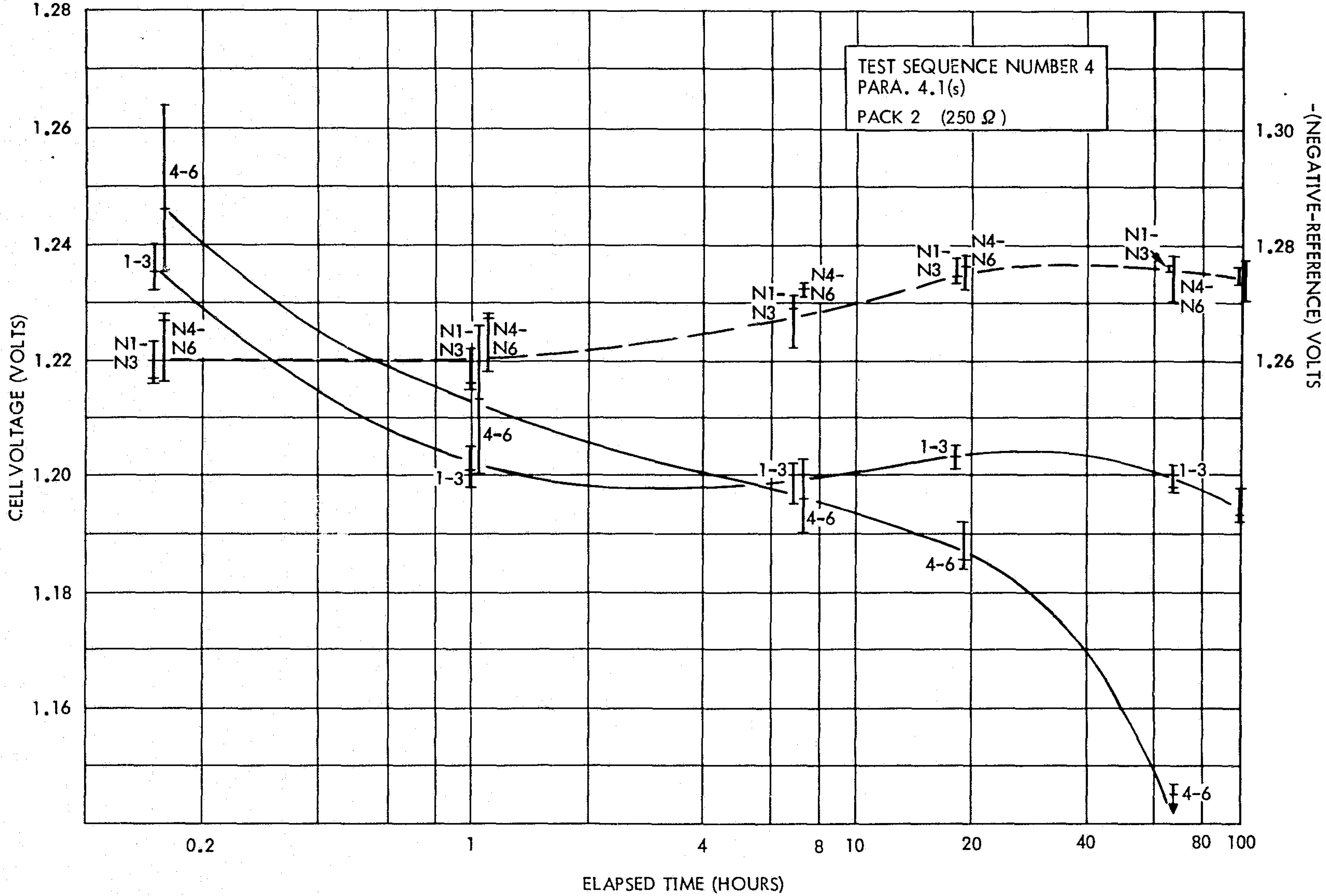

Figure 58. Voltage Decay, Test Sequence No. 5, Paragraph 4.1(s), Pack 2

in Figure 57 that the polypropylene cell voltages showed a shallow minimum around one hour after opening the circuit, then increased gradually by about 15 mV. The nylon cell voltages showed a weak minimum between 1 and 10 hours and a weak maximum at around 24 hours. In Figure 58 the polypropylene cell voltages went through a weak maximum around 24 hours, while nylon cell voltages declined continuously, with the rate of decline increasing rapidly after 24 hours. Neg/Ref voltages increased slightly after the first hour in all these cells, leveling off after about 24 hours. The shapes and slopes of these curves are still distinctly different from those of all the other types of cells tested (Test lots 1 through 4).

During this same period packs 3 and 4 were subjected to a final voltage recovery test, during which the open circuit time was extended to 100 hours. The results are shown in Figures 59 through 63. Note once again the voltages of all these cells were still increasing after 24 hours on open circuit, and those in pack 3 (no external resistors, Figures 59 and 60) continued to increase for 100 hours. The Neg/Ref voltages changed by not more than about 20 mV during these runs.

The voltages in Figure 59 are seen to take many hours longer to rise to say, 1.15 volts, than did these cells after only 16 hours on 0.1 or 0.25 ohms. As the cell voltage changes in this test (Test Sequence Para. 4.1(s)) must be due to changes in the potential of the positive electrode, the slower cell voltage recovery shows that the positive potential returned to its open circuit value more slowly than did the negative electrode to its open circuit value in the earlier tests.

The cell voltage curve for cells with polypropylene separators with 250 ohm resistors attached (Figure 62) is clearly different from that in Figure 60. The former curve is 30 mV lower at 24 hours, and passes through a maximum at around 1.17 volts. The latter feature would be seen only if the open circuit observation time is extended to longer than 48 hours.

Figure 63 shows the data from the same voltage Recovery Test for the three cells having nylon separators and 250 ohm resistors attached. The voltage scale of this plot is much different from that in

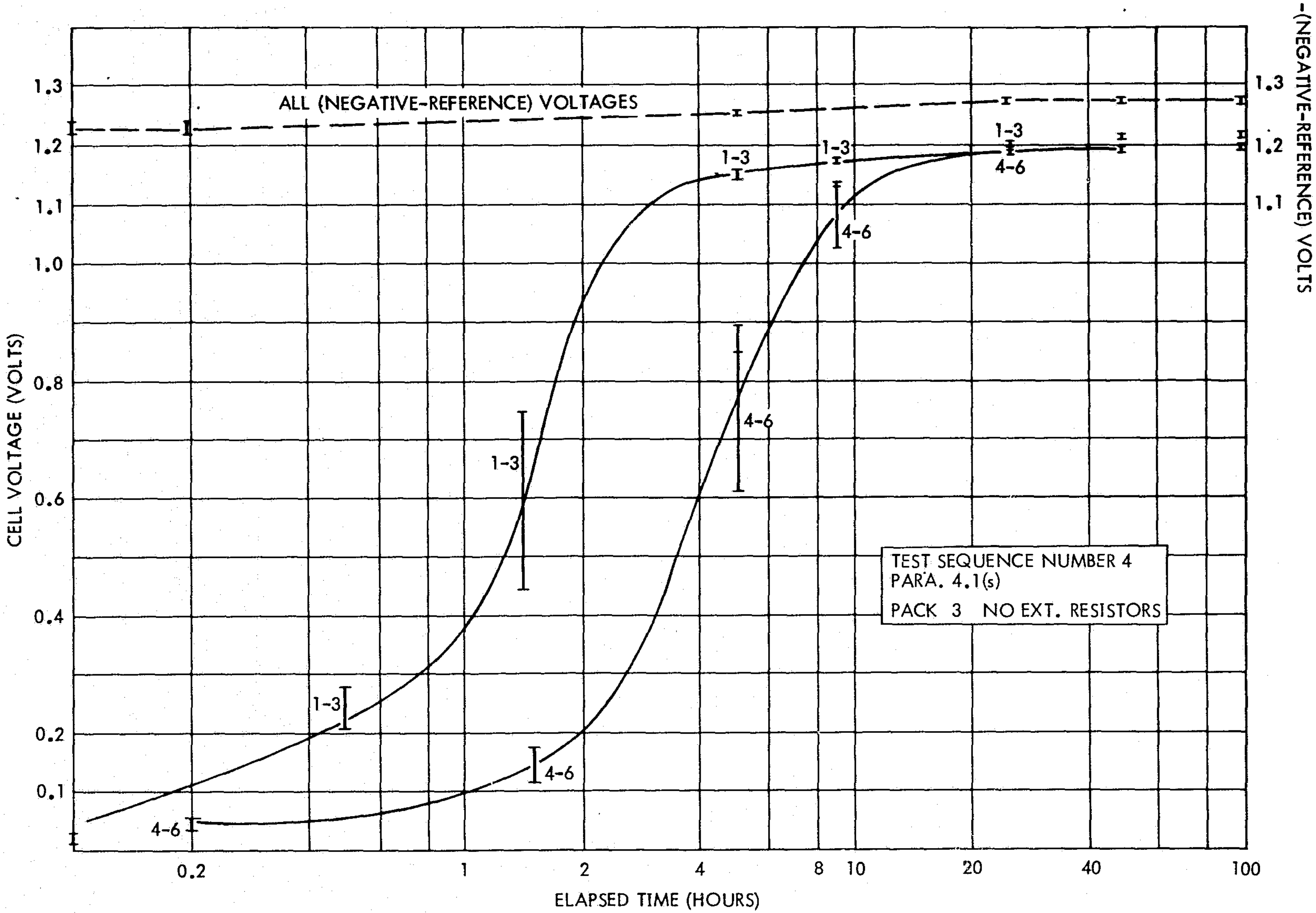

Figure 59. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(s), Pack 3

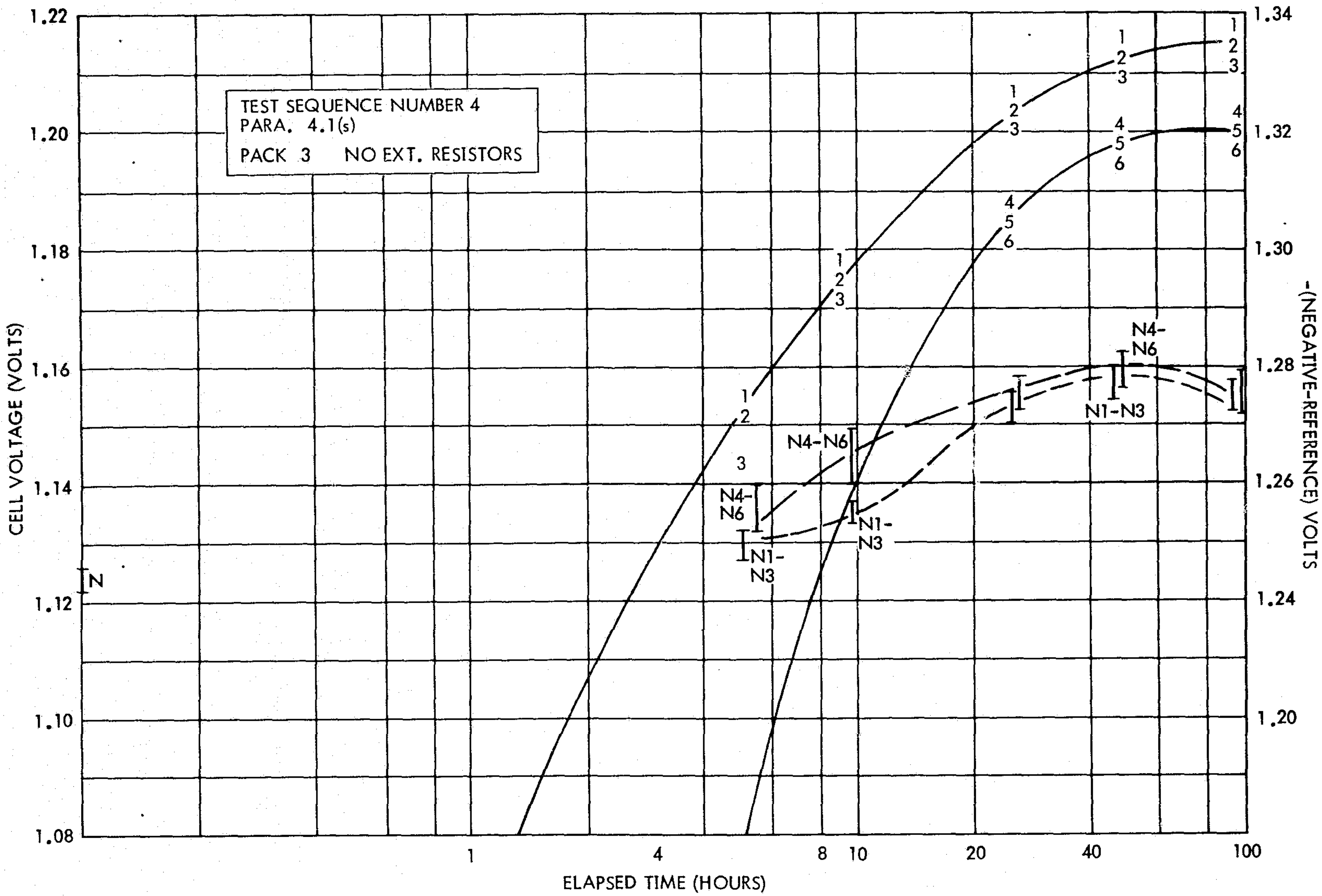

Figure 60. Voltage Recovery, Expanded Plot, Test Sequence No. 5, Paragraph 4.1(s), Pack 3

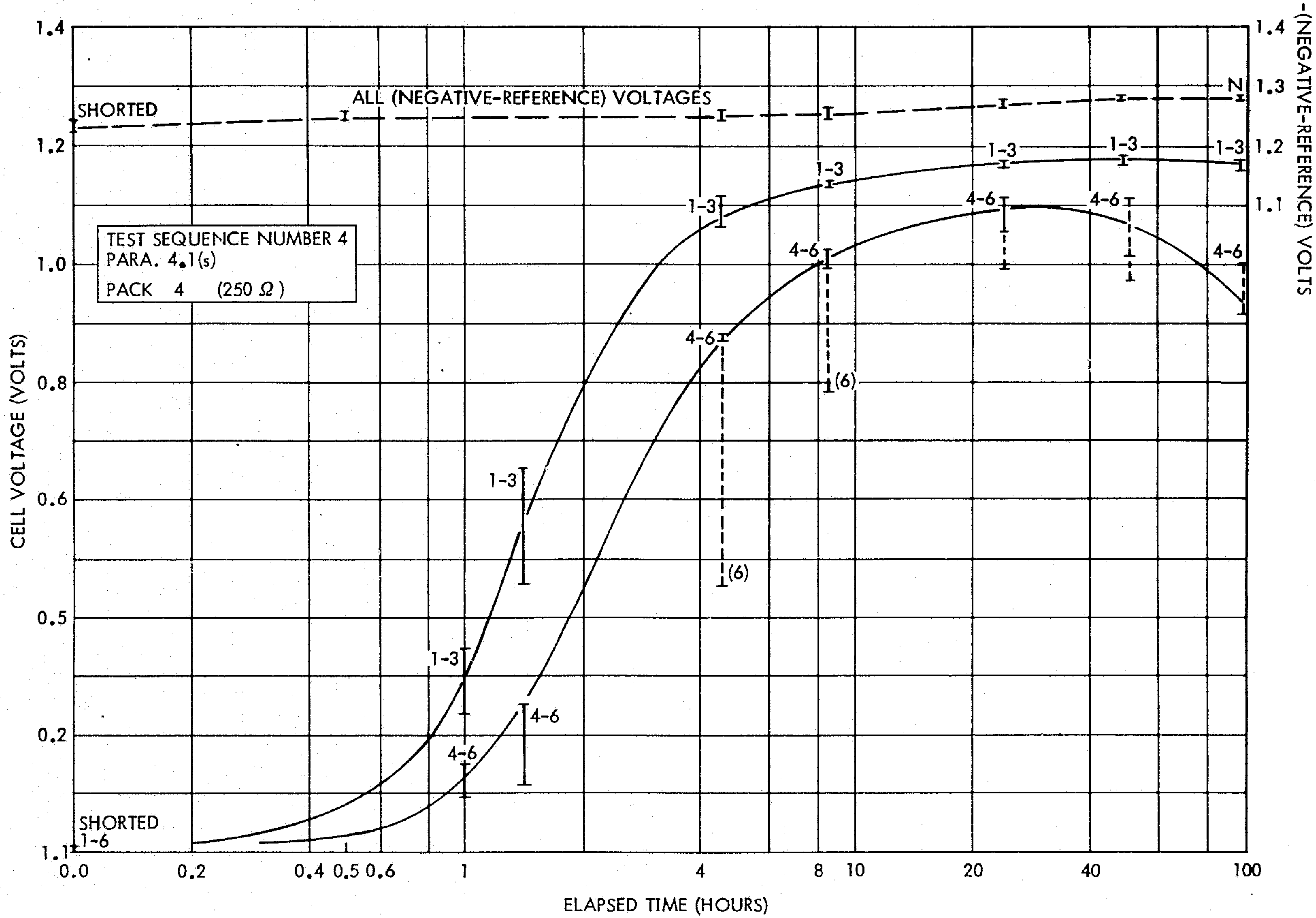

Figure 61. Voltage Recovery, Test Sequence No. 5, Paragraph 4.1(s), Pack 4

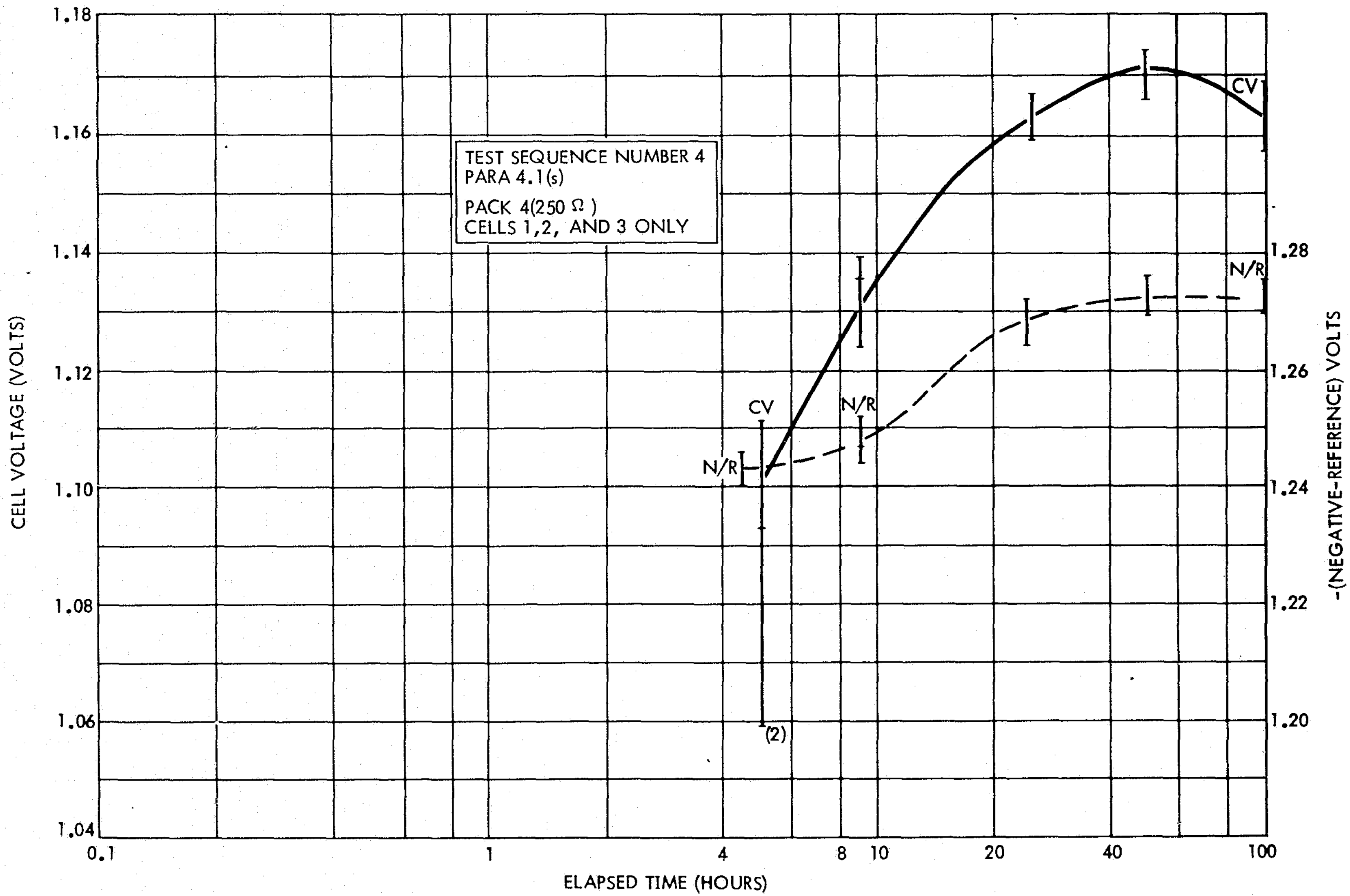

Figure 62. Voltage Recovery, Expanded Plot, Test Sequence No. 5, Paragraph 4.1(s), Pack 4, Polypropylene Separators

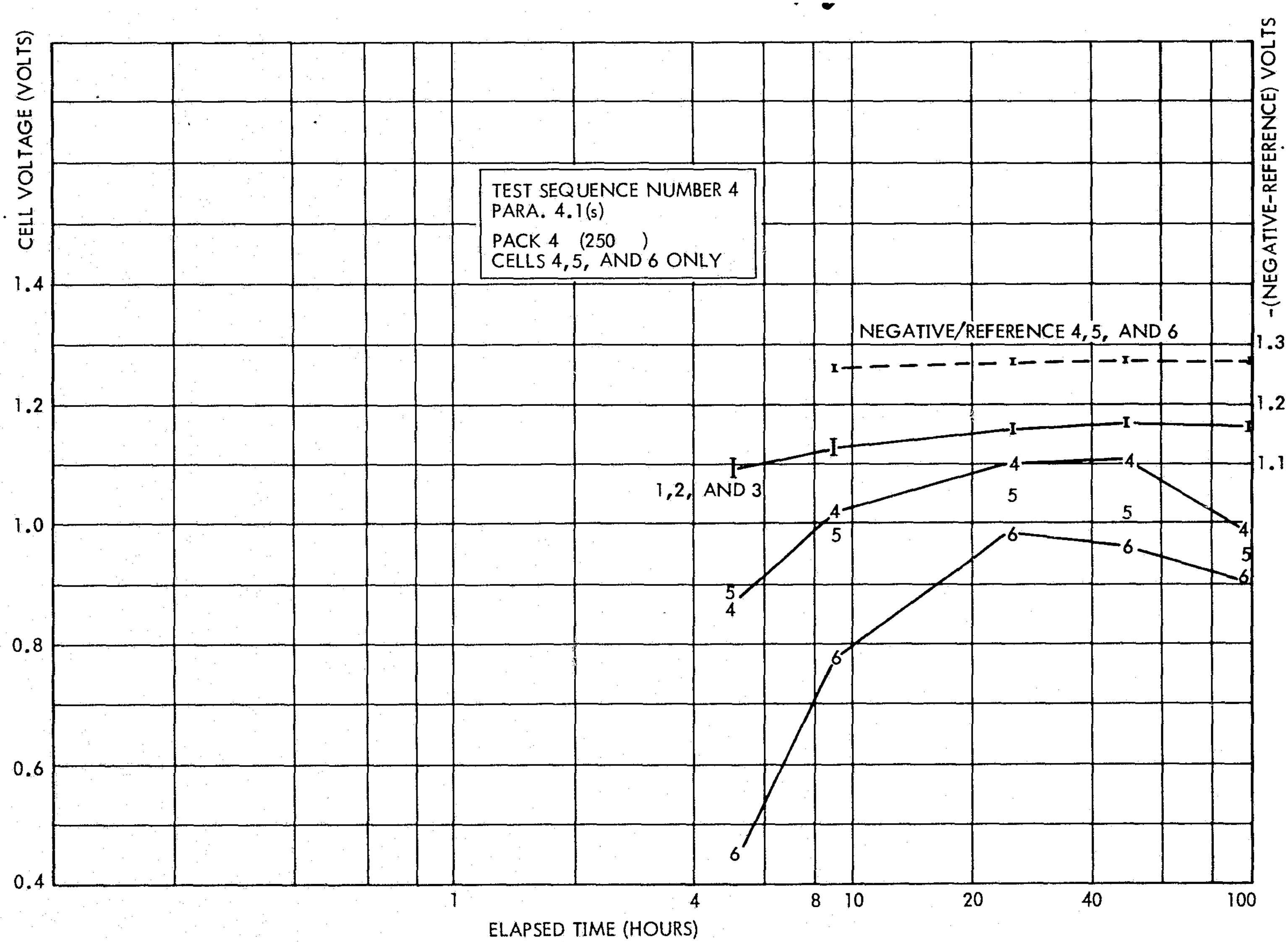

Figure 63. Voltage Recovery, Expanded Plot, Test Sequence No. 5, Paragraph 4.1(s), Pack 4, Nylon Separators

Figure 62. The voltages for the polypropylene-containing cells (Nos. 1, 2, and 3) in this pack are also shown for reference. The difference between the response for the two different types of separators is obviously much grater with the 250 ohm resistors attached than in the absence of an external resistor.

4.3.5.2 Results from Testing 50 ah Cell Packs 5 through 8

The first part of the test sequence for the twenty-four 50 ah cells designated as packs 5, 6, 7 and 8 was the same as that for packs 1 through 4. A composite plot of the open circuit voltage response for the twelve cells having polypropylene separators after removing the 0.1 ohm resistors following the first charge-discharge cycle (Procedure para. 4.2(c)) is shown in Figure 64. The cell voltage may be seen to be entirely controlled by the negative electrode potential here. The response of the cells with nylon separators was quite similar to that shown in this figure.

At the end of the next 0.1 ohm short-down period after a full charge-discharge cycle, all negative electrode potential were close to those of the reference electrodes, indicating that the cells were under negative electrode potential control at this point. The open circuit voltage response of cells 1, 2 and 3 of each pack for the 24 hours following that is shown in Figure 65. Although the voltages of the pack 8 cells (with 50 ohms attached) were noticeably lower, the difference between these cells and those in pack 5 (with no resistors attached) was only 30 mV at 24 hours, and the pack 8 cells were still at about 1.19 volts.

In order to test whether additional discharging would increase sensitivity, 0.1 ohm resistors were put back on all these cells for six hours, i.e., without a charge-discharge cycle) and then again removed. The results for cells 1-3 are plotted for an additional 48 hour stand in Figure 66. It may be seen that the voltages at 24 hours were about the same as before, with pack 8 voltages only about 10 mV lower than before. Data for most of these cells in packs 5, 6 and 7 containing nylon separators were similar to data for the cells containing polypropylene (shown). However, the voltages of two cells in pack 8 and one in pack 6 were in the range of 1.078 to 1.147 at 24 hours, and were down to 0.883 to 1.132 after 48 hours. As these data did not appear to contribute to the study

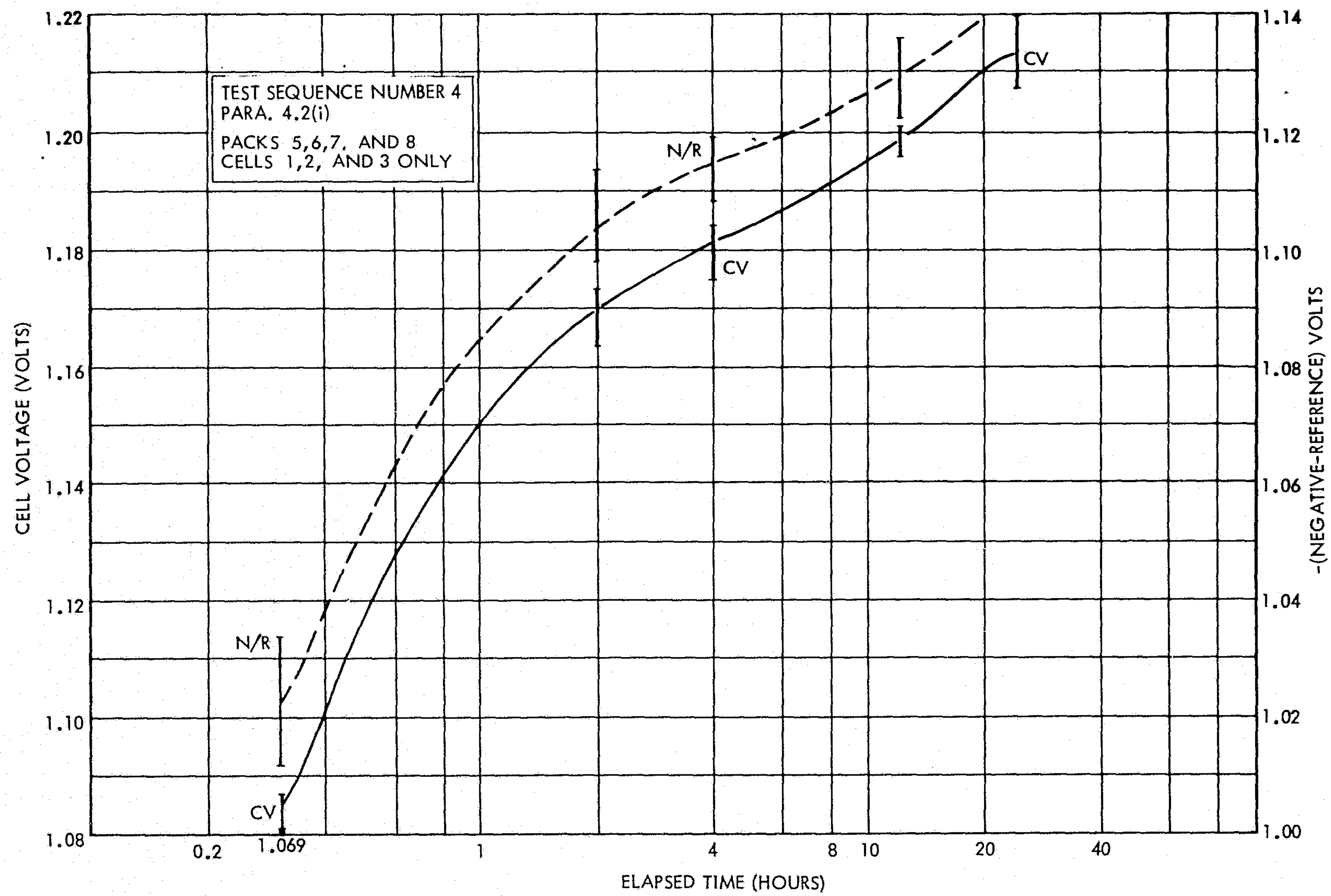

Figure 64. Voltage Recovery, Test Sequence No. 5, Paragraph 4.2(c)

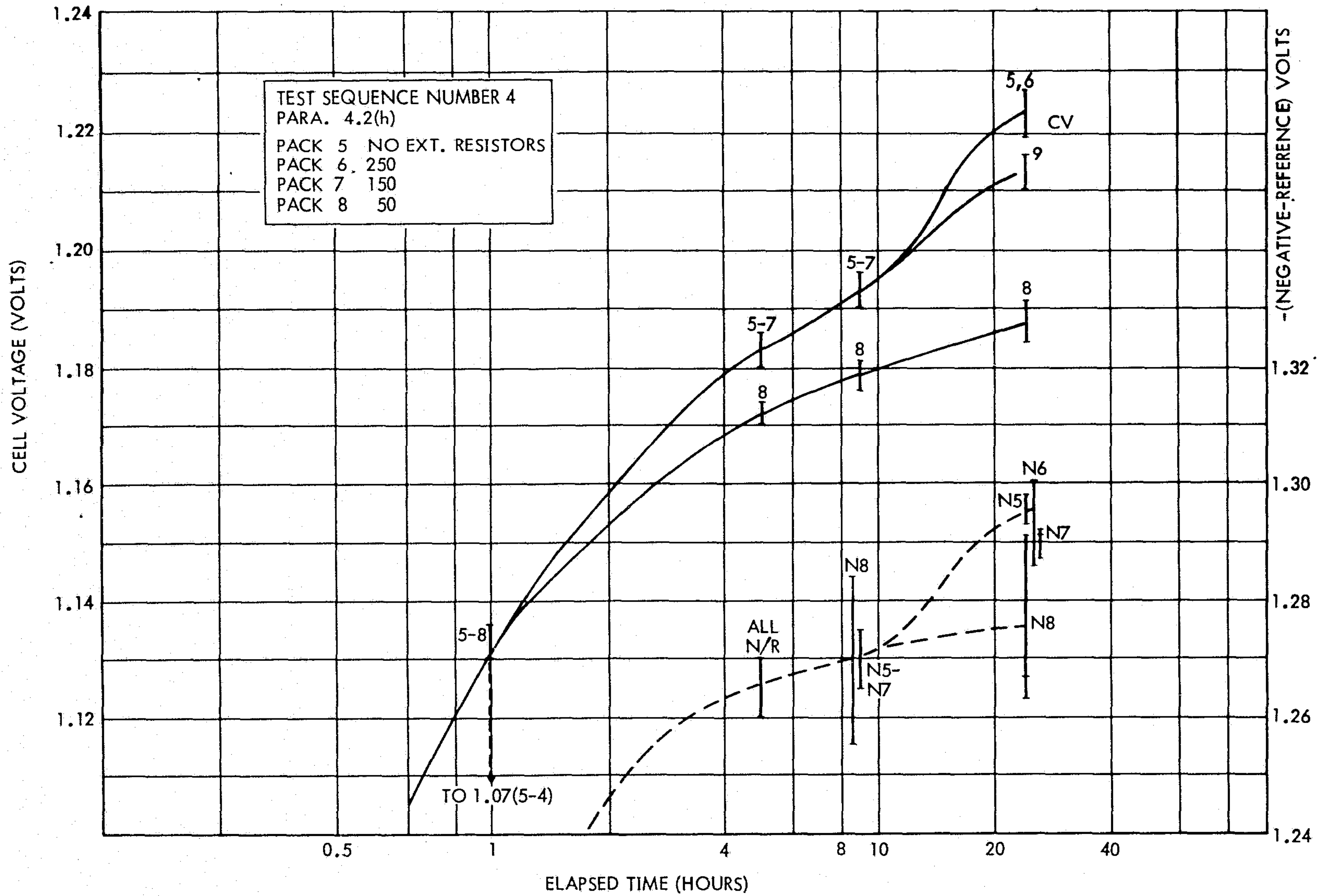

Figure 65. Voltage Recovery, Test Sequence No. 5, Paragraph 4.2(h)

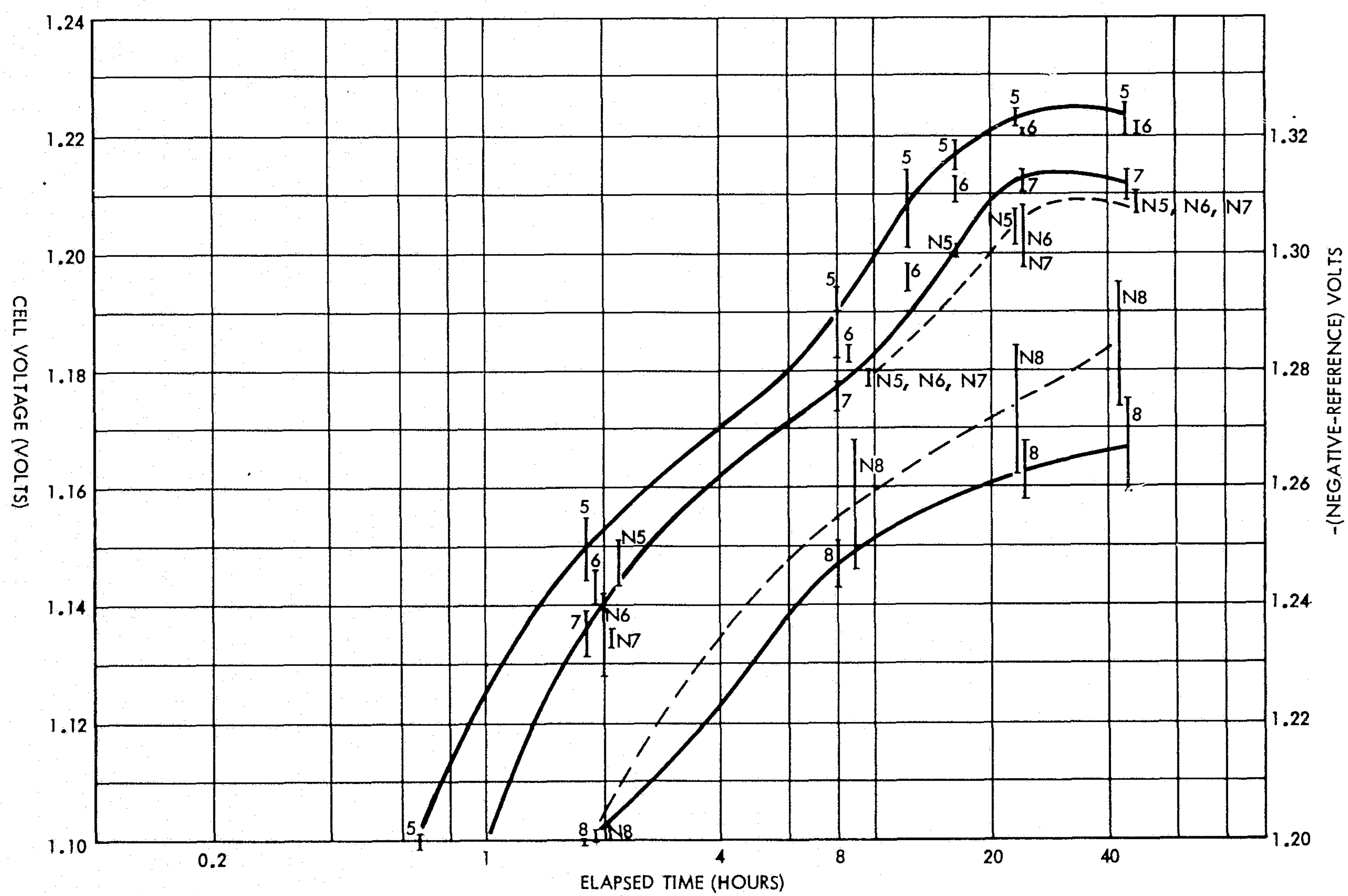

Figure 66. Voltage Recovery, Test Sequence No. 5, Paragraph 4.2(j)

of the test method per se, and comparative data for the nylon vs. polypropylene cells are shown elsewhere, the data for nylon-containing cells during this test were not plotted.

After a subsequent 4 day period on 0.1 ohm resistors followed by a full charge and discharge, 0.1 ohm resistors were attached for 24 hours. During this period the additional high value resistors on the cells were changed to lower values (see Procedure, Para. 4.2(m)) in order to determine the resistance at which the voltage response would be obviously different from that of cells having no resistive path (i.e., that for pack 5 in the previous test). At the end of the 24 hour period on 0.1 ohm resistors, all cell voltages were in the range of 0.2 to 0.3 volts and the Neg/Ref voltages ranged from -0.5 to -0.7 volt in cells 1 through 3 and from -0.5 to -1.23 volts in cells 4 through 6 of each pack.

After the first 3 hours an open circuit following the 0.1 ohm loading the voltages of pack 8, with 10 ohms attached, were about 0.3 volt and decreasing rapidly, while those of the other packs were above 1 volt and increasing. The 10 ohm resistors on pack 8 cells were considered to be too low and were changed to 250 ohm resistors at that point to obtain more useful data. Data obtained subsequently is shown plotted in Figures 67 and 68. The cell voltages for cells 1 through 3 of all packs were closely grouped and describe well defined response curves. Voltages for cells 4 through 6 were widely scattered, with the degree of scattering increasing as the external resistance decreased. The trend of cell voltages of the highest voltage cells in packs 5 and 8 reflected the trend of the negative electrode potential as in other such tests described above. These data, together with those in Figures 58 and 59 indicated that the detectable resistance threshold was about 100 ohms for a 24 hour open circuit stand and about 200 ohms for a 48 hour open circuit stand, following the 24-hour 0.1 ohm stand.

During the next period on 0.1 ohm resistors, the high resistances were again changed, to no resistors on pack 5, and 500, 250, and 100 ohm resistors on packs 6, 7, and 8 respectively. With these resistors in place, capacities were measured and a seven day charged stand was then performed. Voltage data during this stand period are shown in Figure 69.

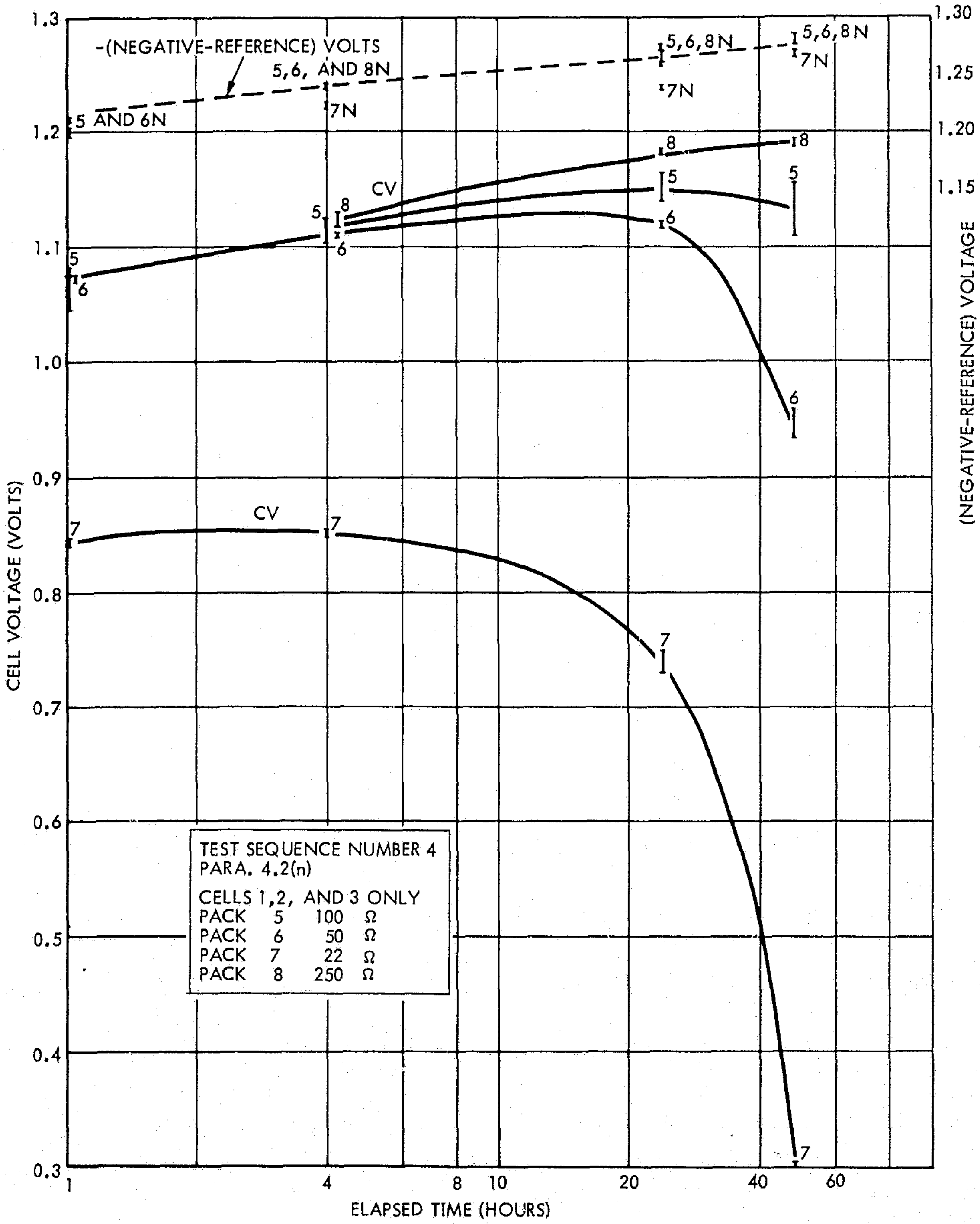

Figure 67. Voltage Decay, Test Sequence No. 5, Paragraph 4.2(n), Polypropylene Separators

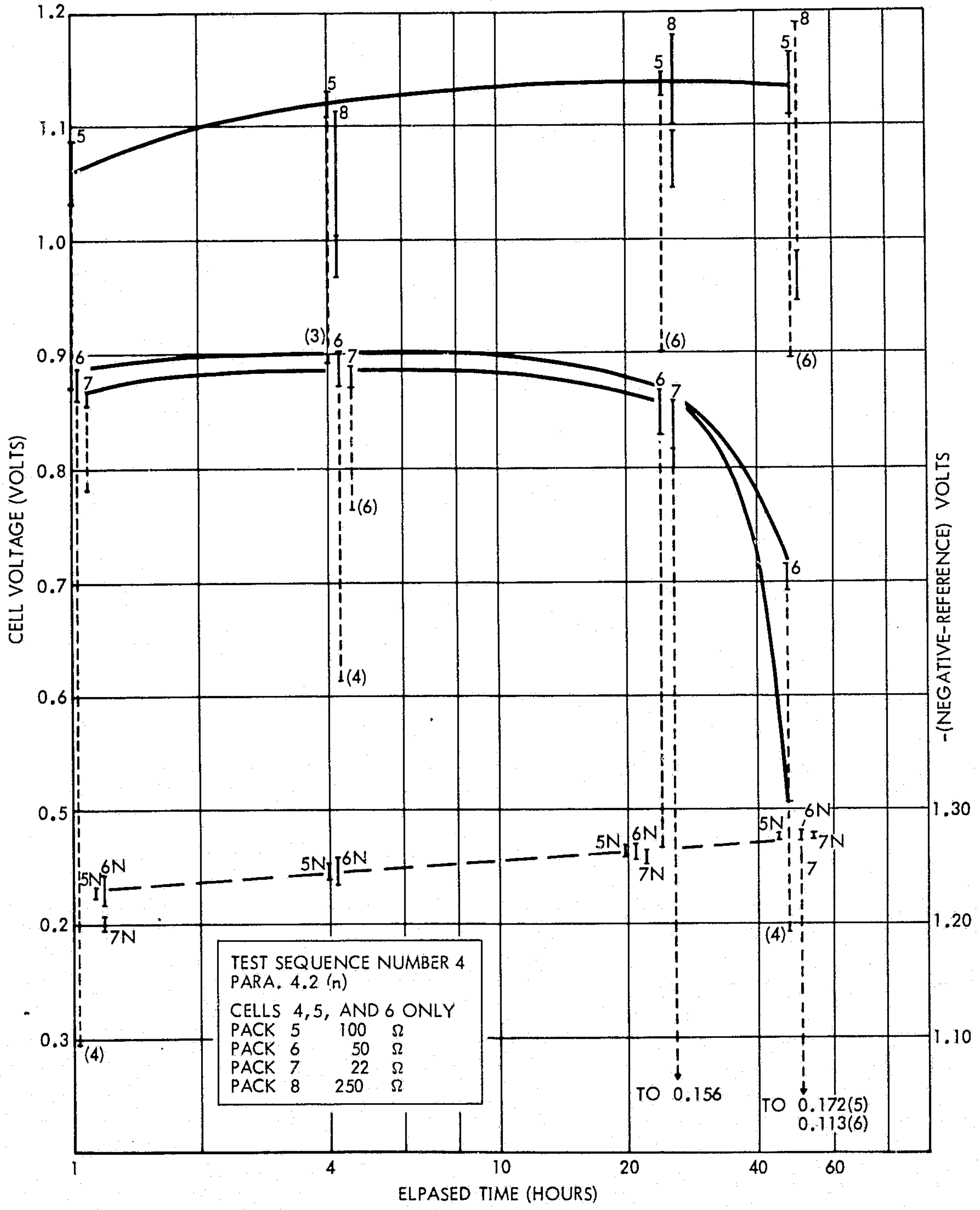

Figure 68. Voltage Decay, Test Sequence No. 5, Paragraph 4.2(n), Nylon Separators

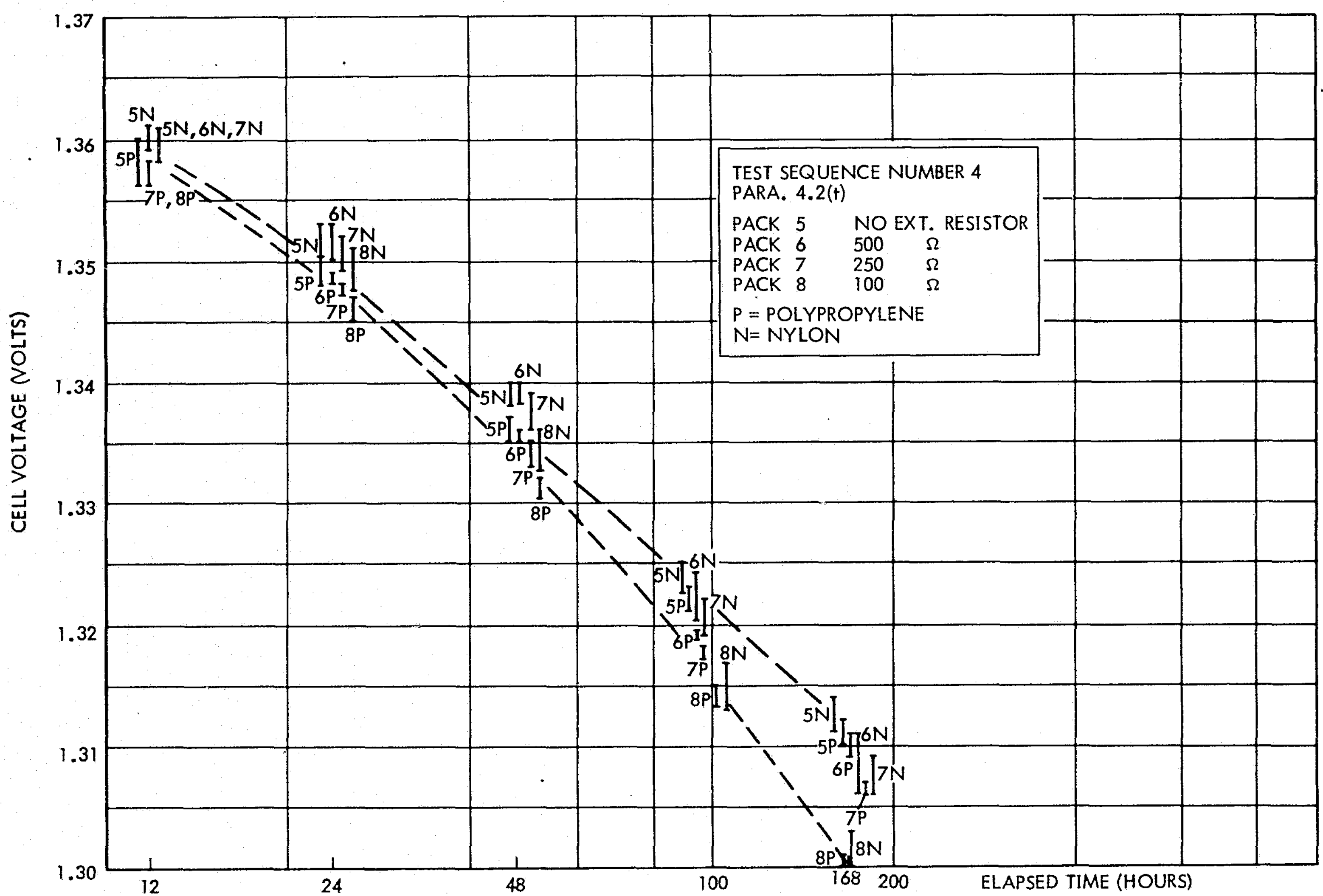

Figure 69. Seven-Day Charged Stand Voltage, Test Sequence No. 5, Paragraph 4.2(t)

It may be seen that the maximum spread in voltages of the six cells in any one pack (all having the same external resistors) was less than 10 mv at all points on the figure (10 to 168 hours on open circuit). Also of interest is the fact that the spread of voltages from the nylon-containing cells was only slightly greater than for polypropylene-containing cells, and the average voltages are higher for the former, in contrast with the results from the other types of tests run. The voltages of the pack 8 cells (100 ohms added) are the only ones that are clearly distinguishable (below) the others at 168 hours.

The cell discharge capacity data from this test are shown in Table 4-4. It may be seen that the capacity retention of the nylon-containing cells (Nos. 4-6) was consistently slightly higher than that of the polypropylene containing cells. There was a slight downward trend in retention as the externally applied resistance decreased, but the differences between the packs shown in Table 4-4 are considered in the "noise level." Hence the resistance practically detectable by capacity loss in this type of test appears to be less than 100 ohms for the 50 Ah cells.

5. SUMMARY AND DISCUSSION

5.1 General Topics

The method of plotting open circuit voltage data as a function of the logarithm of open circuit stand time has been shown to be most useful for the display of open circuit voltage data. By means of this type of plot the voltage data is spread out early in the open circuit periods when voltage is changing relatively rapidly, and the data is condensed toward the end of the open circuit periods when voltage changes are slow. No particular mechanism for the voltage change is implied by this plot.

The open-circuit cell voltages for the 12, 15, and 24 ampere-hour cells during Voltage Decay tests were quite well behaved, in that the voltages always decreased continuously with time following a charge, and the data tended to fall along lines that were either straight or which had small curvature on a logarithmic time scale (except when the cells were shorted

Table 4-4 Capacity Retention Data

		Capacity (Ah)		Before/After (Percent)	Three-Cell Averages	
		Before	After		Cells 1, 2 & 3	Cells 4, 5 & 6
Pack 5	Cell 1	60.9	54.5	89.5		
	2	62.4	55.7	89.3		
	3	60.7	54.0	89.0	89.3	
	4	59.9	55.5	92.6		
	5	60.4	55.5	91.9		
	6	60.8	55.7	91.6		92.0
Pack 6	Cell 1	60.1	53.5	89.0		
	2	60.1	54.0	89.9		
	3	60.1	54.0	89.9	89.6	
	4	62.3	58.0	93.1		
	5	62.3	58.0	93.0		
	6	60.6	55.0	90.8		92.3
Pack 7	Cell 1	60.7	54.0	89.0		
	2	60.7	54.0	89.0		
	3	60.7	54.0	89.0	89.0	
	4	59.6	55.0	92.3		
	5	59.8	55.0	92.0		
	6	60.8	55.0	90.5		91.6
Pack 8	Cell 1	60.9	54.0	88.7		
	2	60.5	53.0	87.6		
	3	60.5	53.4	88.3	88.2	
	4	60.0	54.5	90.8		
	5	61.6	55.0	89.3		
	6	62.5	55.0	88.0		89.4

with relatively low resistors). The cell voltage during recovery from a shorted condition was not expected to be linear on this type of plot, and was not in fact. Yet the log time scale was useful for the display of these data also.

The open-circuit voltage behavior of the 50 Ah cells tested was distinctly different from that of the smaller reels mentioned above. In the 50 Ah cells, after a charge cell voltages at first decreased, then increased, and in the presence of sufficient load, again decreased during open circuit stand. As the negative electrode underwent closely similar changes, this cell voltage behavior is attributed to negative potential control in these cells, as opposed to positive potential control in the smaller cells. This behavior and possible remedial measures are discussed further in later sections.

No reference electrodes were present in any of the smaller cells, so that no accurate measurements of individual electrode potentials could be made in these cells. However, the potential difference between the cell case and the negative terminal was recorded during some of the runs in Test Sequences 1 through 4. Extensive negative electrode potential data was obtained in the 50 Ah cells by means of the built-in reference electrodes, and these data help to explain the difference in behavior observed, as discussed below.

The voltage response of the 50 Ah cells containing nylon separators was consistently poorer and more erratic than that of the same type, size, and vintage of cells made with polypropylene separators. Because of this difference, the data for nylon cells was treated separately from that from polypropylene cells. The data for the 50 Ah cells with nylon separators is discussed further in Section 5.4.

5.2 Application of Results to Test Method Improvement

Because the application of the results is different for the three different tests included in this work, the material relevant to each test is discussed under separate headings.

5.2.1 Application to the Short Term Voltage Decay Test

In general, most aspects of prior history, conditioning, and test performance were found to affect the open circuit voltage response on the short-term Voltage Decay test. The magnitude of the effects on the voltage after 24 hours, was found to be as great as 50 mV, and the variations occurred largely in the range of voltage where the pass-fail criteria currently in use are located, i.e., from 1.15 to 1.20 volts. This finding indicates that when a single-point voltage criteria is used for acceptance of cells, good cells could be rejected unnecessarily unless many variables are carefully controlled. On the other hand, because of a lack of understanding of the effect of such variables, the pass-fail voltage criterion may be set so low that cells with a significant internal shorted condition are accepted. The following sections address these problems in the light of the results of this study.

5.2.1.1 Effects of Prior History and Conditioning

The prior history condition evaluated most extensively was shorted storage. The only form of prior history involving cycling that was included in the experimental program was an Acceptance Test and 30 cycle burn-in sequence performed on new cells, and involving a total of 40 cycles. The effects of other forms of prior history are inferred from the results obtained in this work.

Long-term shorted storage of cells was found to result in failure of almost all cells to meet the usual short-term short test criteria unless the cells were properly conditioned prior to testing (see below). Most cells put on a C/10 charge directly after removal from extended shorted stand did not reach the minimum voltage for adequate charge acceptance within six minutes, and hence could not be expected to give meaningful results. Those cells that did reach an acceptable voltage on charge all decayed much more rapidly than properly conditioned cells.

The length of time on shorted stand that results in the above type of behavior was not established precisely, and is probably a function of the usage prior to the shorted stand. Cells shorted for a year gave the type of response described above; newer cells shorted for several weeks following completion of acceptable testing at the manufacturer responded "normally"

without prior reconditioning, although the open circuit voltages were lower and more scattered than when tested after conditioning. Indications from other data were that results of the short term test begin to become somewhat erratic if the test charge is introduced directly after more than a week or two of shorted stand without an immediately preceding conditioning cycle. Testing of previously shorted cells without conditioning is not recommended, however, as discussed below.

The effect of prior cycling without intervening conditioning is less dramatic and less predictable than that of shorted storage. This is because there are many variables involved in cycling and relatively few involved in the shorted condition. The Acceptance Test and 30-cycle burn-in involved in Test Sequence No. 1 resulted in lowering the open-circuit voltage at 24 hours by 10-20 mV and a small change in the shape of the voltages vs. time curve, but did not appear to alter the sensitivity as determined by consideration of the complete voltage-time plots for cells having various external resistances connected across the terminals. These results were obtained after only a 16-hour period with resistive shorts following a 24-hour open circuit stand after the last of the pre-test cycles, and hence these results are considered to represent non-conditioned cells. No comparable results were obtained for completely conditioned cells. In general, however, because extensive cycling alters the initial states of charge of both positive and negative electrodes, and it has been shown in this study that open circuit voltage response is sensitive to these states of charge, anomalous results may be expected more frequently after cycling if the cells are not conditioned than if conditioning is performed.

If cells or batteries have experienced neither shorted storage nor regular cycling prior to short-term short testing, but instead have been under "random" conditions of intermittent cycling and open-circuit stand, the possibilities for anomalous low-voltages not caused by shorts during the test are considerable. This is in part because the activity of the electrodes tend to become most severely out of balance under these conditions.

It is now clear that if the same pass-fail criteria is applied to all cells tested for shorts, and the prior history affects the readings used to judge acceptability independently of the presence or absence of actual

shorts, then conditioning is highly desirable to render the condition of all cells more alike prior to performing a short test.

5.2.1.2 Conditioning Procedures

No single, cut and dried conditioning procedure is best for all cases, as different prior histories require different forms of conditioning. Cells coming off shorted storage may require at least two or three charge-discharge cycles followed by a period of low-resistance shorting to be conditioned. Heavily cycled cells would appear to be conditioned best by first shorting them down for a number of days, followed by one or two cycles and another resistive discharge.

The effects of different methods of charging and discharging for conditioning were not studied experimentally, but a few comments are in order. After cells have been inactive for some time it is common to use a charge rate less than C/10 for the first charge. If time does not permit a second cycle using a C/10 charge rate, it appears that satisfactory results may be expected after the one cycle provided that the charge period is long enough to allow at least a 100 percent overcharge, and that the subsequent resistive discharge is continued to the point where no cells are negative limited (see below). Better results are to be expected if more than one cycle can be run after a long shorted stand.

If necessary, time may be saved by charging at the C/2 rate during most of the charge input for conditioning cycles, provided the cells are able to accept this higher rate. Figures 1, 3, and 4 show data obtained after a pretest cycle using a C/2 charge rate for 2.5 hours starting from a shorted condition. The lower percentage overcharge appears adequate at the C/2 rate. This higher rate is not recommended for standard procedure because it makes cell conditions difficult to control and is more likely to produce high voltages and pressures that could in turn interfere with the test.

The discharge rate used during conditioning cycles does not appear to be critical in the range from C/4 to C/1. The higher the discharge rate used for the last discharge before the final resistive let-down the higher the state of charge at end of discharge, and the longer the time that will be required to bring the positive electrode to a given state of charge. The

C/2 rate is suitable and convenient in that use of this rate allows capacity to be measured during these cycles at the same rate as is used for most other capacity measurements.

The exact value of the resistance used to bring the cell voltage down after a high rate discharge did not affect the results within a range of 12/C down to 5/C ohms tested. Figure 70 shows the current versus time, calculated from the voltages across the resistors, during the let-down period. Surprisingly, the current through the 1 ohm resistor was greater than that through the 0.25 ohm resistors from a point about one hour into the discharge until 12 hours into the discharge. This is apparently because the higher current through the 0.25 ohm resistors during the first hour reduced the state of charge rapidly during that period. The currents again became equal at about the C/200 rate, and decreased to the C/400 rate at 16 hours. Integration under the curves of Figure 65 shows that 6.2 ampere-hours were discharged by the 1 ohm resistor and 5.6 ampere-hours by the 0.25 ohm resistor over the 16 hour period.

It was also found that it made no appreciable difference whether the resistor was left in place during the entire time prior to the start of the following charge, or whether the resistors were replaced by dead shorts for part of the time after the voltage had been reduced to a few tenths of a volt. This result would be predicted if the current is indeed diffusion-current limited as indicated above. Thus, there appears to be no technical reason why the resistors first attached need to be replaced by dead shorts between cycles.

The last cycle performed prior to the injection of the test charge and open circuit stand is a special case, in that all aspects of that cycle may be expected to affect the open circuit voltage response more strongly than those of previous cycles. Thus, special care is necessary to make sure to conduct this last cycle in the same way for all cells and from test lot to test lot.

This is especially true of the final resistive let-down. Results from this study show that 16 hours is not enough time for this step, regardless of what value of resistor is used (including dead shorts for the last part of the time) to assure best results in this type of test for all cells that may be

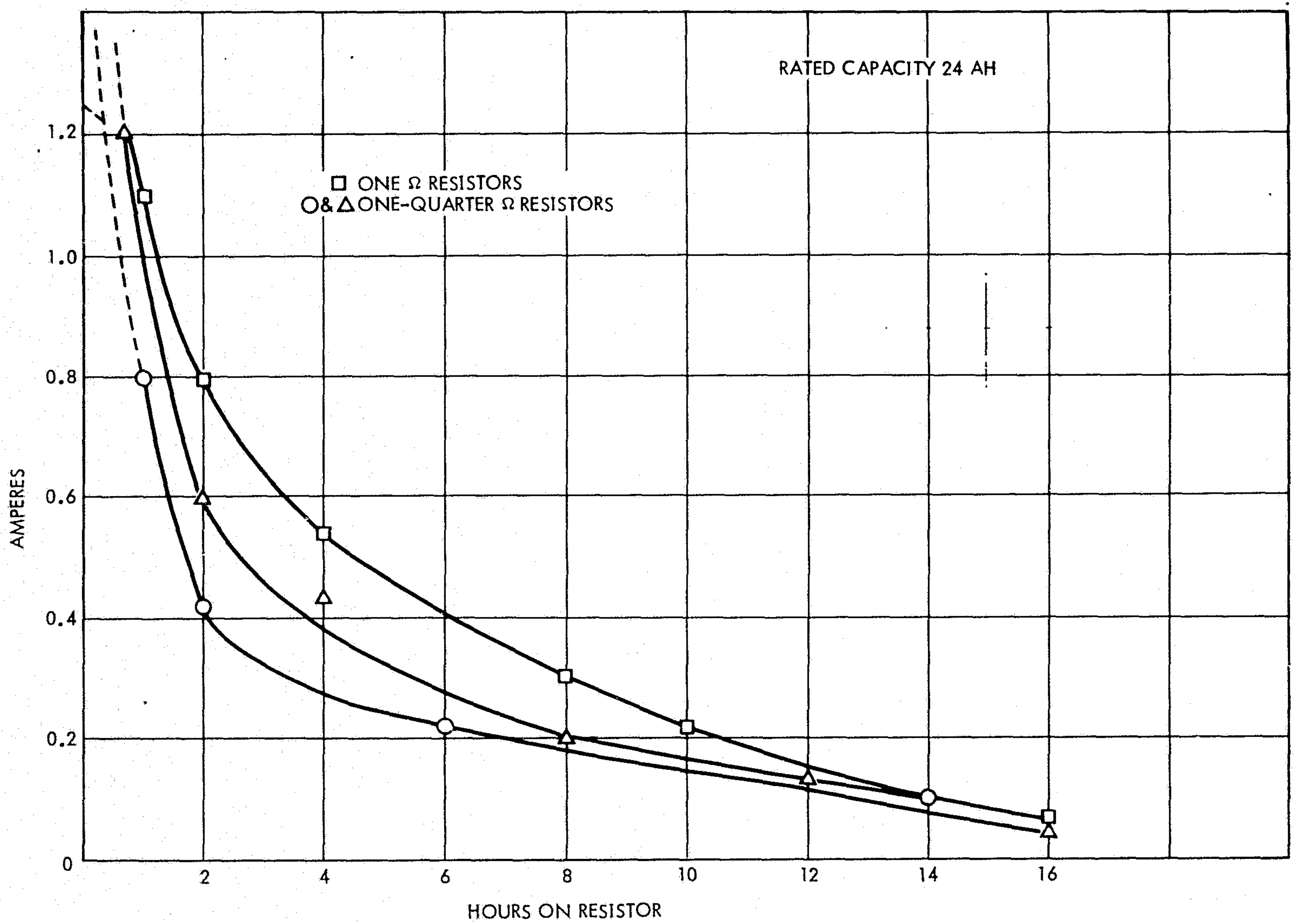

Figure 70. Current vs. Time on Low Resistance During Let-down

encountered. This is because when the voltage of cells is controlled by the potential of the negative electrode instead of by the potential of the positive electrode when the cell is first put on the resistor, considerably more than 16 hours may be required to discharge the positive electrode to the point where its potential becomes controlling. As discussed elsewhere, the latter condition is more desirable for best results from this test. In the study about 32 hours were required for a group of cells that were originally negative limited to become positive limited. Longer times may be required by other cells depending on the severity of negative limiting, i.e., on how much charged positive capacity remains at the end of the C/2 discharge to 1 volt. Because of the close relationship between the conduct of the last (conditioning) cycle and the performance on the subsequent open circuit test, it appears advisable to make the prior cycle and subsequent let-down period a part of the procedure proper, and hence to bring that cycle under close control.

5.2.1.3 The Test Charge

It has been shown here that the charge rate used for the brief test charge injected for the short-term short test is not critical over the range from C/10 to C/2, for the same ampere-hour throughput. Contrary to expectation, the charge acceptance was no greater at the higher limit than at the lower limit of this range. The reason for this result is not known and was not pursued. As the C/10 rate is as effective as others tried, and is easier to control and measure, the C/10 rate is preferred.

An ampere-hour throughput equivalent to about one percent of cell capacity was found to give the best compromise between sensitivity to resistive loads on the one hand and susceptibility to interference by factors not related to shorting on the other. In this study the one percent throughput was produced mostly by charging at the C/10 rate for six minutes. Five minutes of charging at C/10, as called for in many procedures, should give essentially the same results.

A comparison of the plots of cell voltage versus log time in this report with those in Figure 7 shows that, for a one percent (1%) ampere-hour charge, the cell data fall along lines that are straighter than that for an isolated nickel-oxide electrode in the region beyond eight (8) hours on

open circuit. Several possible reasons for this may be suggested. First, the true state of charge of the positive electrode in the cell is probably greater than 1 percent after a 1 percent charge, in spite of the fact that the charge acceptance has been shown to be far less than 100 percent, because the positive electrode is very difficult to discharge to zero state of charge. On the other hand, because the electrodes used to generate the data in Figure 7 had been extensively reversed with liberation of hydrogen before being charged, part of the charge may have gone to react with absorbed hydrogen, thus resulting in the actual state of charge being less than the percent charge shown.

A review of all the end-of-test-charge voltage data generated during this study indicates that subsequent open circuit behavior was always normal when the end of charge voltage was ≧1.32 volts; and behavior was often erratic when the end of charge voltage was less than 1.32 volts. As failure to reach this voltage appeared to be more a function of inadequate conditioning than of an internal shorted condition, results for cells not reaching this voltage should be considered invalid, and the procedure should be repeated on such cells before passing judgement. Of course, if the cell has a low-resistance internal short, the on-charge voltage may not reach 1.32 volts for that reason, and a retest would show a similar response the second time.

5.2.1.4 The Open Circuit Stand Period

Consideration of all the plots of open-circuit voltage over periods varying from 48 to as long as 100 hours leads to the conclusion that, in general, a 24-hour stand time is not long enough for best results. Greater sensitivity and freedom from interference can be achieved if 40 to 48 hours are allowed (after a one percent charge). With positive limited cells, the resistance across the cell that could be reliably detected increased in proportion to the open circuit stand time allowed. For best results with negative limited cells the minimum useful stand time is determined by the time required for the potential of the negative electrode to stabilize after opening the circuit. Even after a 40 hour resistive discharge prior to injecting the test charge, the potential of the negative required about 24 hours to become constant in one group of cells tested (as shown in Figures 57 and 58).

It is possible, of course, to perform this test on negative limited cells without extending either the prior short-down period or the open circuit period beyond the 16-24 hours and the 24 hours, respectively, normally prescribed at this time. Doubtless, many such cells have been tested this way. But the results of this study show that under these conditions the drift of the negative electrode potential during the open circuit period can cause the cell voltage response to bear little relation to the rate of discharge of the cell over a wide range of resistance loads, and by so doing decrease the sensitivity of the test well below the inherent capability of the method.

On the other hand, the results of this test became less favorable again as the stand time after a one percent charge was extended beyond 48 hours. With charge throughputs greater than one percent the optimum stand time was extended correspondingly beyond 48 hours. However, these combinations of greater charge and longer stand time did not appear to offer any advantage, and would only consume more time.

One way to minimize the time required for the test and yet assure best results would be to include reference electrodes in the cells and observe the negative to reference voltage during the let-down period, stopping when the negative is seen to have levelled off at its normal unpolarized potential. However, this would require adding a reference electrode to the internal structure of all cells, and hence is not practical in most cases. An alternative is to monitor the voltage between the negative terminal and the case. Even though the case half-cell potential may drift, this drift is likely to be small compared to the shift in potential of the negative electrode of the order of 1 volt when the cell goes from being negative limited to being positive limited (e.g., see Figure 56). At the same time the voltage of the negative with respect to the case changes sign (from positive to negative) when this transition occurs, and hence the event is usually easy to identify.

5.2.1.5 Sensitivity

The sensitivity of the method is defined for the purpose of this discussion as the minimum load (i.e., maximum resistance) across the terminals that can be unequivocally detected by analysis of the voltage data.

In this study, the effect of externally applied resistances was detected by comparing the voltage versus time curves for cells with external resistors attached with the curves for other cells of the same type vintages and history that had no resistors attached. If the curves were essentially the same for the two sets of cells, the effect of the resistance in question was considered undetectable. When the curves diverged in a reproducible manner and to a significant extent, the resistance was deemed detectable.

Under the best operating conditions the factors that determined the magnitude of the difference between curves that was significant was the spread of voltages of the cells with the same resistance attached, including no resistor, and the variability from run to run. As the scope of this project did not permit a quantitative statistical analysis of the variance, "significant" differences were estimated by inspection of the plots.

As pointed out earlier, the sensitivity of the short-term voltage decay test, as observed in this study, was a function of many variables. When the test was performed with the proper conditioning of the cells to avoid spurious responses, and when sufficient time was allowed for the final resistive discharge when cells were negative limited, the following sensitivities were obtained as a function of open circuit stand time:

Rated Cell Capacity (Ah)	Sensitivity (ohms) After a stand time of:	
	24 Hrs.	48 Hrs.
15	500	1000
24	250	500
50	100	250

Thus the sensitivities in terms of resistance were inversely proportional to cell capacity, as might be expected. The figures for 48 hour sensitivity can be normalized with respect to cell capacity by means of the expression:

$$\text{Sensitivity (48 h)} \cong \frac{1.25 \times 10^4}{C} \text{ ohms,}$$

where "C" is numerically equal to the rated capacity of the cell in ampere-hours.

The sensitivity of this method when applied to negative limited cells (without first converting them to a positive limiting condition) is less. Comparison of Figure 49 with Figure 48 shows that (for the cells with polypropylene separators) the difference in voltage between 50 Ah cells with 100 ohms attached and those with no resistors was about 10 mV at 24 hours and 20 mV at 48 hours. These differences are not considered sufficient for reliable detection in the presence of the variability apparent in Figure 49, and hence up to 48 hours the sensitivity must be 50 ohms or less for this size cell. Note that this is one-fifth the sensitivity obtained by testing after the cell was positive limiting.

In this case continuation of the open-circuit stand beyond 48 hours resulted in a voltage behavior in that time frame that sharply increased the sensitivity. As shown in Figure 49, between 48 hours and 100 hours the cell voltage decreased relatively rapidly with the 100 ohm load whereas (Figure 48) the voltage of the cells with no added load increased slightly. Thus, taking both the difference in slope and the difference of 40 mV at 100 hours into account would indicate a sensitivity of the order of 200 ohms. This is comparable to the value for 48 hours stand time obtained on positive limited cells.

It is interesting that the total amount of test time required to achieve this latter order of sensitivity is about the same, whether or not the cells are conditioned to produce positive limiting before injecting the test charge. In one case the time is used to discharge the cell more completely; in the other case the extra time is spent on open circuit. Of the two, the prior approach is technically more sound and is recommended.

5.2.1.6 <u>Acceptance Criteria</u>

5.2.1.6.1 <u>Single-Point Voltage Criteria</u>

Procedures currently in use call out a single-point voltage requirement for acceptance of cells, which states that the cell must be at or above a specified voltage at 24 hours after putting the cell on open circuit. This voltage ranges from 1.16 to 1.19 in different procedures reviewed. A lower limit of 1.16 volts is too low for an absolute single-valued criteria when applied to relatively new cells, as shown by the following analysis.

Table 5-1 contains a compilation of the average voltage of several groups of six cells, after 12, 24 and 48 hours on open circuit, determined on this study using a 1 percent test charge input. Only data for conditioned cells are included. The average of these data for each cell classification is shown in Table 5-2.

Table 5-1. Voltage Data for Voltage Decay Tests

Cell Classification	Figure No.	6-Cell Average After O. C. Stand Time of: 12 Hours (v)	24 Hours (v)	48 Hours (v)
New/Tested	11	1.223	1.221	1.219
	18	1.210	1.205	1.201
	23	1.216	1.213	1.210
Medium/Stored	38	1.217	1.210	1.203
Old/Stored	31	1.200	1.188	1.176
	33	1.222	1.212	1.193
	35	1.204	1.198	1.190

Table 5-2. Average Open-Circuit Voltages as Function of Stand Time

Cell Classification	Stand Time: 12 Hours	24 Hours	48 Hours
New/Tested	1.216	1.213	1.210
Medium/Stored	1.217	1.210	1.203
Old/Stored	1.209	1.199	1.186
All Inclusive	1.214	1.207	1.200

The values from Table 5-2 for "New/Tested" and "Medium/Stored" cells are shown plotted as curves 1 and 2 in Figure 71. It is seen that the data form straight lines of different slope, intersecting at 14 hours. Dotted line 1L indicates the lower limit of the voltage of individual cells corresponding to average Curve 1.

Point "A" shows the location of 1.16 volt end-point at 24 hours. It may appear that the scale used unduly exaggerates the difference between Curves 1 and 2 and Point A. However, the scale shown is the same as that used for most of the other plots of Voltage Decay data in this report, and is used here to facilitate visual comparison.

As implied by the figure, the results of this study show that the lowest average voltage at 24 hours that should be considered normal for new cells is about 1.20 volts, and that any new cell passing through Point A is clearly abnormal. Furthermore, the study shows that about the only way that the voltage of a relatively new cell properly conditioned and tested could go as low as 1.16 volts within 24 hours on open circuit (i.e., following Curve 3 in Figure 71) would be for the cell to have an internal short of the order of $0.25 \times 10^4/C$ ohms resistance (unless it is subject to the special problem with nylon separators seen in some cells). Although this level of internal shorting resistance may be considered tolerable for some applications, the method has been shown to be inherently capable of detecting higher resistances (lesser shorting loads) and should be used to do so as far as possible. Inspection of Figure 66 indicates that a 24 hour voltage of 1.19 volts should not be too low for high-quality new/tested cells. A more quantitative statistical analysis of the data will be required to determine the limits in terms of the standard deviation.

It may be argued that a low allowable lower limit is necessary because older cells are tested by this method and thus the criterion must take into account the general trend (as seen in Table 5-2, for example) toward lower open circuit voltages in older cells. Without considering the implication of this trend for internal shorting, it seems clear that, with the data now available, there is no longer any reason to sacrifice the effectiveness of the test for new cells by using acceptance criteria tailored to old and possibly partially shorted cells. Instead, it is recommended that different

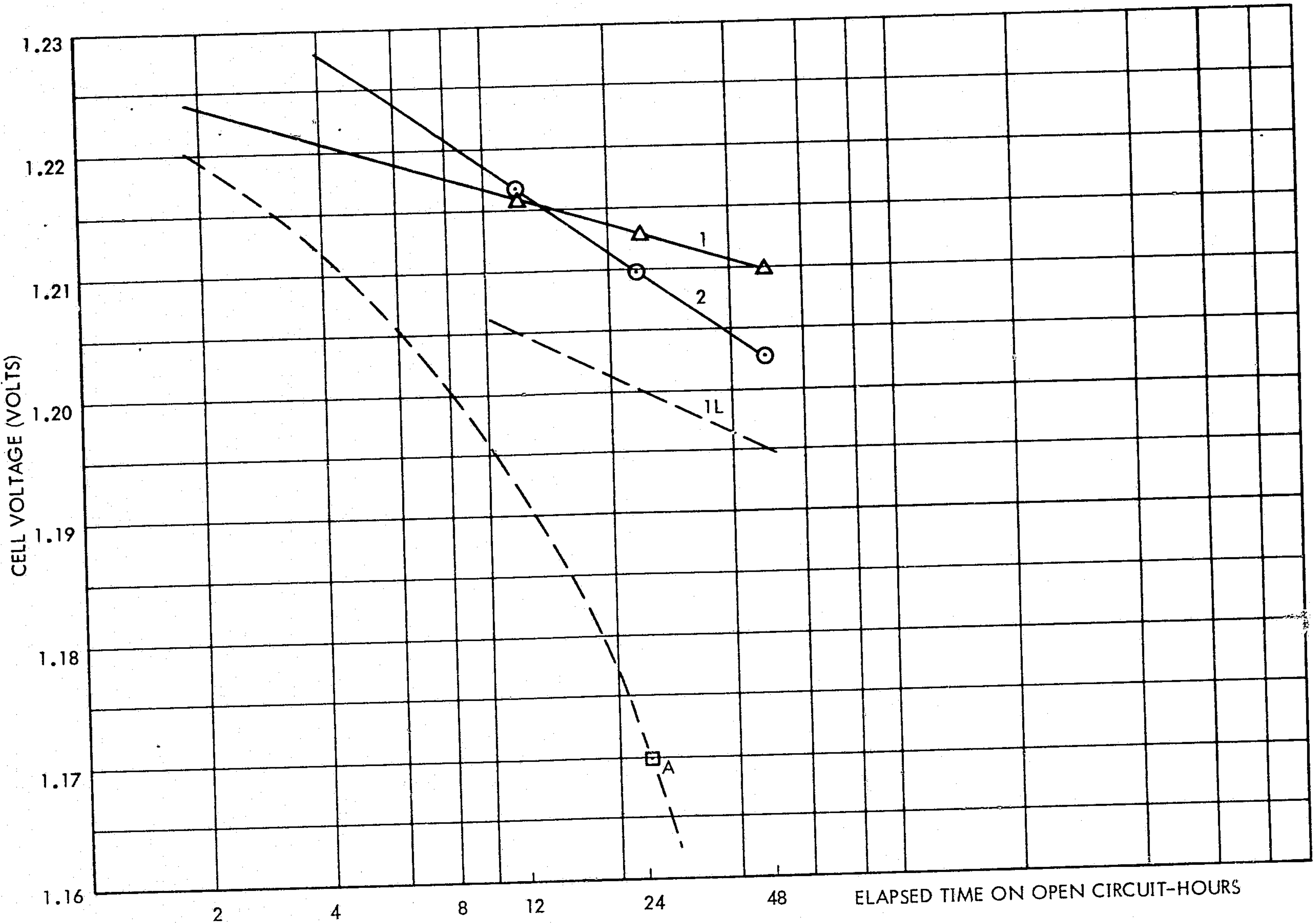

Figure 71. Average End-Voltage Data

criteria be applied to different age-groups of cells, with a higher cut-off voltage used for new cells than for old cells.

It may also be argued that a lower cut-off voltage is needed to accommodate the normal range of variation in conditions and details of testing techniques encountered in practice. In reply, it is pointed out that the present study considered the effects of a wide range of variables, and the only combinations that resulted in low voltages on new/tested cells were those which resulted in a marginally low state of charge during the open circuit period and accompanying erratic loss of cell voltage due to phenomena other than shorting. In order that the test be meaningful, it must be carried out in a manner that avoids such erratic response. When this is done, the average voltage for new/tested cells may be expected to be near Curve 1 of Figure 71, and hence the minimum acceptable voltage should be consistent with that curve.

5.2.1.6.2 Improvement of Acceptance Criteria

One of the barriers to more effective use of open-circuit voltage testing for screening cells is the fact that most procedures call for taking only a single reading, at 24 hours, during the open circuit stand, and that only this single data point is used to judge the cell. One of the practical disadvantages of this approach is that if for any reason the reading is not or cannot be taken at the 24 hour point (plus or minus a tolerance), or if the readings taken are obviously erroneous or questionable, then the entire test is invalidated and must be repeated with the associated expense and loss of time. A more basic objection to the single point determination is that it provides only a minute fraction of the performance information that can be obtained during the test period. More voltage data would increase useable sensitivity and assist in interpreting marginal or anomalous results.

This study has shown that the taking of more frequent voltage readings during the open-circuit period is practical and does not interfere with the test, provided that measuring equipment used has sufficiently high input impedance. Most modern electronic voltmeters easily satisfy this requirement.

When multiple voltage readings versus stand time are available from the test, a variety of more discriminating criteria may be applied to judge the condition of cells. These include:

a) Conformance to two or more sets of voltage-time data pairs, i.e., V_1 at t_1; V_2 at t_2; V_3 at t_3; etc.

b) Slope of the voltage-time curve. Different slope criteria may be applied at different times.

c) Slopes in combination with voltages, as a function of stand time.

d) Slopes of the curve, i.e., is it a straight line, or does it curve downward or upward.

Table 5-1 contains sets of voltage-time data for three different stand times which could be used as criteria for averages. Note that the differences reading across any one line are close to being constant. For the new/tested cells the difference between any two readings ranges from 0.002 to 0.005 volt. If one were to allow a maximum difference of 0.005 volt, for example for a 12-to-24 or 24-to-48 hour interval, then the slope on a semi-log scale would be:

$$\Delta V/\Delta(\log t) = 0.005/\log 2 = 0.005/0.30 = 0.0017$$

This would mean that the voltage could not differ by more than 0.0017 volt between any two points differing by a factor of two in open-circuit stand time.

The overall slopes of the curves drawn through multiple data points give additional information. The data from good-quality cells without shorts tend to give voltage data lying close to a straight line from about 2 hours to beyond 48 hours on open circuit, whereas inadequately charged cells give S-shaped curves and cells with significant shorts give lines that curve downward continuously. The simultaneous application of all the above criteria, which is possible when a series of readings is taken on open-circuit, should greatly enhance the usefulness of these open-circuit tests.

5.2.2 Application of Results to the Voltage Recovery Test

5.2.2.1 Effects of Prior History and Conditioning

In general, the above discussion of the effects of prior history and conditioning on the Voltage Recovery Test applies to the Voltage Recovery Test also. It was seen that, without a charge-discharge cycle immediately preceding the test, no cells may be expected to pass the test after more than a few days on low-resistance or shorted stand. For this reason it is advisable to include the prior cycle as an integral part of the test procedure, rather than considering that cycle as only part of a pretest conditioning.

Performance of this preliminary cycle can have an undesirable effect on some cells. As observed in this study, this cycle can convert previously positive limited cells into negative limited cells. Thus, if cells that are marginal in active precharge are charged at C/10 for 24 hours, discharged at C/2 to 1 volt, and let down on a 6/C ohm resistor, they may be negative limited at that point even though they were positive limited prior to the charge. Inasmuch as this prior cycle is essential to the Voltage Recovery Test, the possibility of having a negative limited cell when the circuit is opened must be recognized. Guidelines for dealing with such cells are discussed below.

5.2.2.2 Final Pre-Open-Circuit Discharge

The method of conducting the final let-down prior to opening the circuit was shown to be more critical for the conduct of this test than it is for the Voltage Decay Test. This is probably because in the latter test the test charge injected tends to "normalize" the state of charge from cell to cell, whereas no such normalizing is performed in the Voltage Recovery Test. This makes the open circuit voltage recovery characteristics after shorting dependent wholly on the effects of the prior charge-discharge cycle(s) and final let-down. This variability may be compounded by the effect of non-constant negative electrode potentials if negative-limited cells are involved.

Figure 37 shows a response that was typical of non-shorted positive-limited cells following a 16 hour resistive let-down and a 4 hour shorted period. The cell voltages required between 2 and 3 hours to rise to over

1.10 volts, and the rate of increase became small after about 6 hours, with the voltage continuing to increase slowly for the remainder of the open circuit stand time. This region of gradual increase began above 1.15 volts for most cells tested.

The condition of positive limiting prior to opening the circuit is indicated by the fact that the negative-to-case potentials were around -0.7 volts before the low resistors were removed[1]. These potentials became only slightly more negative when the loads were removed, and changed further only by a relatively small amount during the open circuit period. However, because the case was the only available "reference electrode" in these cells, the precision of this data is subject to question. Nevertheless, they are consistent with other observations and data, indicating that the half-cell potentials of the cases were quite constant during these tests, and hence the changes observed in the negative-to-case potentials truly reflect changes in the negative electrode potentials.

The response of non-shorted cells that are negative limited at the time when the low resistance load is removed is illustrated by Figures 39 and 43. Note here that the cell voltages rose to 1.1 volts in less than 30 minutes, that the negative electrode potentials in most cells were near

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[1] As the cell voltages (V_c) were near zero, and cell currents were very small just before removing the low resistors, we may write

$$(E_p - E_{case}) - (E_n - E_{case}) = V_c \cong 0$$

Where E_p and E_n are the positive and negative electrode potentials respectively. Then, substituting -0.7 volts for $(E_n - E_{case})$, and solving for $(E_p - E_{case})$ gives:

$$(E_p - E_{case}) \cong -0.7 \text{ volt}$$

Hence, the positive electrodes are polarized to the normal negative electrode potential.

zero, (with respect to the nickel-oxide reference electrode[1]), and that the negative electrode potentials changed by 0.5 to 1 volt during the first few hours after the circuit was opened. Inspection of the curves shows that the initial rise in cell voltage was due largely to a change in the negative electrode potential, whereas in positive limited cells the initial rise is due entirely to a change in the positive electrode potential.

In negative limited cells the cell voltage continues to be strongly a function of the negative electrode potential for at least the first 48 hours on open circuit during voltage recovery. This is shown clearly in Figures 40, 41, 44, 64, 65, and 66, in which an expanded scale is used in the region above 1.1 volt. These relatively large changes taking place in the negative electrode potential tend to obscure changes in the positive electrode potential that are related to a discharge path.

For comparison, the voltage recovery response of a group of 50 Ah cells (initially negative limited) that had been allowed to stand with 0.25 ohm resistors for 40 hours after a C/2 discharge (see Figure 56 for a representative plot of negative potential behavior during the short-down) is shown in Figure 59 for a 100 hour open circuit stand. Note here the similarity between the cell voltage vs. time curve and that in Figure 37, and the similar flatness of the negative electrode potential characteristic. An expanded plot of that portion of the response above 1.08 volt is shown in Figure 60. Comparison of Figure 60 with Figure 44 for a negative limited

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(1) The reference electrode in these (50 Ah) cells is at a half-cell potential that is about 0.5 volt positive with respect to the case $E_{ref} - E_{case} = 0.5$ Volt).

As $E_n - E_{ref} \cong 0$, then the potential of the negative with respect to the case was

$$E_n - E_{case} = (E_n - E_{ref}) + (E_{ref} - E_{case}) \cong +0.5 \text{ V}$$

Thus, the negative (and positive) electrode was at a potential about equal in magnitude but opposite in sign relative to the case compared to the positive limited cell in Figure 34.

cell shows how different the response was in the region of voltage used for acceptance of spacecraft cells.

The benefit from sufficiently discharging certain cells is apparent from the above. On the other hand, once the cell becomes positive limited, or on cells that are already positive limited at the end of a 16-24 hour letdown, additional discharge can have the effect of causing non-short-related low voltage responses. Therefore, monitoring of the potential of the positive or the negative electrode in the cell in order to determine which electrode is limiting is advisable for best results with the Voltage Recovery Test.

5.2.2.3 Sensitivity and Open Circuit Stand Time

The only meaningful measurements of sensitivity of the Voltage Recovery Test during this study were made on the 50 Ah cells. The few measurements made on the smaller cells were made under non-optimum conditions and so the data is not included here. Thus, the results discussed below were obtained on cells that were negative limited or which had been made positive limited by an extended short-down. Results for the latter will be discussed first, followed by those for negative limited cells.

Comparison of voltage data over a 100 hour open circuit stand period for positive limited cells with and without 250 ohm resistors attached (Figure 61 vs. Figure 59, and Figures 62 vs. Figure 60), indicates that, for polypropylene separators, the shapes of the response curves were similar up to 48 hours, but the voltages for the cells with the added resistors were consistently 40 mV below those of cells without resistors. As the spread within any one group was less than 10 mV from 12 hours on, the sensitivity at 24 hours was estimated to be between 250 and 500 ohms on this test.

The difference between the two curves remained the same from 24 to 48 hours on stand, so that no changes in sensitivity resulted from the 24-hour to 48-hour data. However, between 48 and 100 hours the voltage of the cells with 250 ohm resistors started going down, while that of the cells without external resistors continued to rise slowly. This difference in behavior increases the ability to differentiate between these two sets of

data, thus the sensitivity at 100 hours was estimated to be greater than 500 ohms.

Sensitivity was substantially lower when the Voltage Recovery Test was performed on negative limited cells (without first converting them to a positive electrode potential control condition). Although the overall response curves for 50 Ah cells with and without 100 ohm resistors were very similar in appearance (see Figures 43 and 45), inspection of the expanded plots in Figures 44, 46, 65, and 66 leads to the conclusion that the useable sensitivity was 50 to 100 ohms after 24 hours on open circuit. This is one-fifth the sensitivity achievable with positive-limited cells.

Only one test involving an open-circuit stand time longer than 24 hours was run on negative-limited cells. As shown in Figure 66, the voltages leveled off after 24 hours and maintained their relative positions up to the end of that test at 48 hours. Hence no advantage of extending the time was apparent in this case.

The reason for the difference in sensitivity between the positive-limited and the negative-limited cells was not pursued experimentally; however, the following hypothesis is suggested. When a cell is positive limited[(1)] at the time the external low resistance or short is removed, the cell voltage begins and continues to be almost entirely a function of the positive electrode potential. Therefore, if a short is present, the current through the short will cause the positive electrode potential to be lower than otherwise because of the instantaneous polarization effect (which is small) and because the positive capacity is being further discharged as time passes. Hence the open-circuit positive potential corresponding to the decreasing state of charge is decreasing with time.

[(1)] The term "positive-limited" is used here to denote the condition where the positive electrode is nearly completely discharged and/or the positive electrode potential is sensitive to cell current, whereas the negative electrode potential is insensitive to cell current and remains near its unpolarized value.

When the circuit is opened on a negative-limited[1] cell, the potentials of both electrodes may change rapidly at first, then more slowly as time passes. After being strongly polarized, the negative electrode potential appears to drift for a long time (toward more negative values) before it becomes constant. Data in the figures show that more than 24 hours are required for this process. And because a negative change in the negative electrode produces an increase in cell voltage, this drift opposes any change that may be produced by changes in the positive electrode. In addition, the residual undischarged positive capacity is much greater in a negative limited cell, and thus this capacity will take a correspondingly longer time to discharge through any shorting path resistance, prolonging the occurrence of any detectable change in cell voltage. In view of this argument and the demonstrated lower sensitivity obtained on negative-limited cells, it is recommended that such cells not be tested for shorts without treatment to make them positive limited.

5.2.3 Application of Results to the Charged Stand Test

No experimental work was done to examine the effect of prior history or conditioning of cells on the results of the Charge Stand test. Two tests involving about the same prior history, and using the same procedure for measurement of capacity prior to and after the 7-day open circuit stand were performed. Each of these tests involved some cells with various resistors attached to calibrate the sensitivity of the method.

The voltage data for the older, 15 Ah cells indicate a sensitivity of less than 500 ohms, estimated as 300 ohms. This is consistent with the small difference in capacity retention found (84 vs. 87 percent) between cells with 500 ohm resistors attached and those with none attached.

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[1] The term "negative-limited" is used here to denote a condition where the potential of the negative electrode is sensitive to current while the potential of the positive electrode is not. This condition may arise because the electrochemical activity of the precharged negative material has degraded to the point where the usable negative capacity is less than the usable positive capacity. If this occurs the positive capacity will not be completely discharged when the cell is discharged to near zero volts at any appreciable rate. The resulting residual charged capacity then stabilizes the positive potential.

The voltage data for the test performed on the 50 Ah cells indicate a sensitivity of 100 ohms for this size cell. The capacity retention data indicate a sensitivity somewhat less than 100 ohms. When normalized to rated cell capacity, the figure from the 15 Ah cell test may be expressed as 4500/C ohms, whereas that from the 50 Ah cell test would be expressed as 5000/C ohms. Thus these two tests gave essentially the same level of sensitivity as a function of cell capacity.

5.3 Comparison of Test Methods

In this section the three open-circuit test methods investigated in this study are compared for susceptibility to interference, ease of interpretation of data, sensitivity, and time required.

5.3.1 Susceptibility to Interference

The three methods are increasingly subject to interference in the order (a) charged Stand Test, (b) Voltage Decay Test, and (c) Voltage Recovery Test. The forms of interference referred to are the variable effects of prior history and details of immediately prior cycling and conditioning procedures. It is expected that this order would be maintained under average operating conditions in the presence of the precautions recommended in this report.

5.3.2 Interpretation of Results

The voltage data from the Voltage Decay Test and the Charged Stand Test applied to positive limited cells are more easily interpreted in terms of shorting resistance than are data from the Voltage Recovery Test, because the former lend themselves better to calibration. Whereas voltage decay data tend to lie along fairly straight lines on a log time plot, voltage recovery data show a pattern in which the slope changes almost constantly with time. Some of the difficulties encountered with voltage recovery data in this study were undoubtedly due to lack of control of all the variables affecting the results. More effort will be needed to bring this method fully under control.

5.3.3 Sensitivity

Estimates of the sensitivity to be expected from the three methods under average working conditions, using the methods as recommended herein, are shown in Table 5-3. These figures have been adjusted somewhat from the figures for optimum conditions described earlier in the text, to account for normal variations.

Table 5-3. Expected Average Sensitivities*

Open Circuit Stand Time (Hrs)	Voltage Decay	Sensitivity, in ohms	
		Voltage Recovery	Charged Stand
24	$0.5 \times 10^4/C$	$1 \times 10^4/C$	--
48	$1 \times 10^4/C$	Not defined	--
168	--	--	$0.25 \times 10^4/C$

*For positive-limited cells

5.3.4 Time Required for Testing

Although the open circuit stand time is much shorter for the Voltage Decay and Voltage Recovery Tests, than for the usual (7 day) Charged Stand Test, the total time required when starting with non-reconditioned cells and allowing 48 hours for open circuit stand, is the same. This is shown by the following typical schedule for a complete Voltage Decay Test:

Operation	Time Required (Hrs)
Low Rate Charge	40
Discharge	4
Let-Down	16
C/10 Charge	24
Discharge	4
Let-Down	40
O.C. Stand	48
	176

If, on the other hand, the short testing is done in the course of other testing involving the proper kind of cycling, the time differential for the Voltage Decay Test would be only half of that shown above.

5.4 Anomalous Results with 50 Ah Cells

As pointed out previously, a marked difference was seen in the open circuit voltage response during Voltage Decay and Voltage Recovery testing between the 50 Ah cells containing polypropylene separators and those containing nylon separators. The former type gave what was considered normal response while the latter type showed a high percentage of relatively low voltages, as if they contained significant shorts. Yet the response of these cells on the Charged Stand test was fully equivalent to, if not better than that of the cells containing polypropylene. This and other performance data indicated that these cells did not contain resistive shorts.

It is clear that the pseudo-shorting effect was associated somehow with the nylon separators, as none of the otherwise identical cells made with polypropylene separators showed any such behavior. In view of the fact that the 24 Ah cells used in Test Sequence No. 1 were of a similar design, were made by the same manufacturer, and were made using nylon separators, one might expect to see some similar behavior in these cells. However, as the data presented in Section 4.3.1 for these cells indicates, they showed no sign of this effect.

Experimental investigation of this effect was outside the scope of this study. However, some inquiry resulted in the accumulation of the following possibly relevant pieces of information:

- Cell manufacturers have noted sporadic high percentages of short test failures, which they have attributed to excessively "tight packs," i.e., where the plates are thicker in aggregate than normal, thus compressing the separator more than normal. The manufacturers profess not to know why a tight pack should act this way.

- Cells which pass short testing during screening at the cell level often fail the same test after they are built into batteries. In most such cases the force on the face of the cells is higher in the battery, and often this force is not as evenly distributed, as it is during cell testing.

These data indicate that the effect may be due to the close approach of positive to negative plates. If so, then the same effect should be seen in similarly compressed cells made with polypropylene separators, yet no such effect has been observed in this study or elsewhere as far as is known.

In view of this, it is postulated that the effect may be due to an acceleration of the rate of reaction of charged nickel active material with separator material, where the accelerated rate is produced by the closer contact resulting from the higher pressures. To account for the fact that this pseudo-shorting problem is sporadic and seems to affect some lots of cells and not others, it is postulated that the molecular weight and chemical stability of nylon separator material may vary from separator lot to separator lot, and even from point to point within one separator lot. If so, then the combination of normal variations in plate thickness, separator thickness and density, cell compression, and separator material chemical composition could result in many of the short test anomalies observed. This problem needs to be investigated further in order that it not degrade the potential utility of open-circuit voltage tests.

6. CONCLUSIONS

Based on the results of this study, the following conclusions are drawn:

1) Shorted storage up to several years causes only a small decrease in the voltage level during open circuit tests, provided cells are properly conditioned after storage. Shorted storage in itself does not cause internal shorting.

2) Cycling of the type and mount (up to 40 cycles) normally performed during Acceptance Testing, burn-in, and screening, can cause a small decrease in the voltage level relative to that for new cells, but this degree of cycling should not cause internal shorting in properly designed and manufactured cells.

3) Conditioning prior to open-circuit testing is beneficial to the results of all tests, and is essential after some forms of prior history. The most effective form of conditioning varies with the prior history.

4) Results of the Short Term Voltage Decay Test are relatively insensitive (a) to charge rate in the range from C/10 to C/2 for a given ampere-hour input, and (b) to the ampere-hour input in the range from 0.5 to 2 percent of cell capacity, provided that the cells are properly conditioned. Reproducibility increases and sensitivity decreases as the input increases. A one percent input gives best overall results.

5) A cut-off voltage of less than 1.19 volts at 24 hours for new cells is not justified by the data. Hence, cells showing lower end voltages are suspect.

6) The usefulness and achievable sensitivity of both the Voltage Decay Test and the Voltage Recovery Test can be increased by extending the open-circuit stand time from 24 hours to 48 hours or longer, provided that voltages are measured periodically during the stand time.

7) The Voltage Recovery Test is more sensitive to immediate prior history and conditioning procedures than the other tests, and the data from this test is more difficult to interpret quantitatively.

8) With sufficient care, the Voltage Recovery Test can be more sensitive than the short-term Voltage Decay Test by a factor of two in 24 hours. After 48 hours on open-circuit the sensitivities of these tests are about equal.

9) All things considered, the (short-term) Voltage Decay Test is preferred for general application.

10) The Seven-Day Charged Stand Test is a factor of 2 to 4 less sensitive than the other two types of tests, but is more immune to prior history effects and other interferences.

11) The taking of multiple voltage readings during open-circuit stand periods provides data that greatly increases the usefulness of the tests over that achieved by measurement of only a single point after 24 hours. A plot of the data on a log time scale is a useful tool for interpretation of results.

12) The condition in cells wherein the cell voltage is controlled by the negative electrode potential (rather than by the positive electrode potential) at the point when the circuit is opened, can seriously interfere with the detection of internal shorts. This condition can be detected and eliminated by simple procedures.

13) New cells with nylon separators can behave during Voltage Decay and Voltage Recovery tests as if they contained internal shorts. The cause of this behavior is not known and bears further investigation.

7. RECOMMENDATIONS

7.1 GENERAL RECOMMENDATIONS

Based on the results and conclusions from this study, the following recommendations are made:

a) All three of the open-circuit test methods included in this study should be retained as having merit, pending the accumulation of additional data under more controlled conditions that may more clearly indicate which, if any, is a superior test.

b) Procedures for the Short Term Voltage Decay Test and the Voltage Recovery Test should be upgraded to include specific requirements for temperature control; for pressure applied to cell faces during testing; for conditioning; and for the taking of data periodically during open circuit stand. Any go/no-go decision should be based on consideration of a number of voltage vs. time readings, rather than on a single such reading.

c) The role of excessive mechanical pressure and of nylon separator material in causing low open circuit voltages should be investigated in order to exploit the full potential of open circuit test methods.

d) Recommended guidelines for improving short test procedures are provided in Appendix A.

7.2 RECOMMENDED IMPROVED PROCEDURE FOR VOLTAGE DECAY SHORT TEST

1. Condition cells as follows:

   a) For cells that have been on shorted storage for more than a few days: Perform one or two cycles by charging at the C/20 to C/10 rate for 20 to 40 hours, discharging at the C/2 rate to 1 volt, then applying resistors of 12/C ohms or less for 16 to 40 hours.

b) For cells that have been cycling and have not been recently shorted, discharge and apply resistors of 12 12/C ohms or less for several days. Then perform one cycle as in 1(a) above.

2. Charge cells at the C/10 rate at 20 - 23°C for 5 or 6 minutes. Verify that the voltage is at least 1.32 volts at end of the charge.

3. Allow cells to stand for 48 hours on open circuit at 20 - 23°C. Measure cell voltages once each hour for the first four hours, then once each four hours thereafter.

4. Plot voltages versus log of open circuit stand time in hours. Consider the entire curve in judging the results. Determine degree of fit of data to one or more straight lines. Non-linear, downward-curving trend indicates a significant internal short. Compare curves with those from cells with known load resistors to calibrate data.

7.3 RECOMMEND IMPROVED PROCEDURE FOR VOLTAGE RECOVERY SHORT TEST

1. Condition cells as follows:

   a) Same as for Voltage Decay Test.

   b) Same as for Voltage Decay Test.

2. Charge cells at the C/10 rate at 20-23°C for 24 hours.

3. Discharge cells at the C/2 rate to 1 volt.

4. Apply resistors having resistance of 12/C ohms or less for 40 hours, or until it has been determined that the potential of the positive electrode is close to that of the normally charged negative electrode.

5. Remove the resistors and allow the cells to stand on open circuit for 48 hours. Measure cell voltages each hour for the first four hours, then each four hours thereafter.

6. Plot the data as a function of time and consider the entire curve in judging the results. Compare curves with those from cells with known resistors to calibrate results.

7.4 RECOMMENDED IMPROVED PROCEDURE FOR SEVEN DAY CHARGED STAND TEST

1. Condition cells as follows:

   a) Same as for Voltage Decay Test

   b) Same as for Voltage Decay Test

2. Charge cells at the C/10 rate at 20-23° for 24 hours.

3. Put cells on open circuit for one hour.

4. Discharge at the C/2 rate to 1 volt. Record ampere-hours discharged.

5. Apply resistors having resistance of 12/C ohms or less for 16-24 hours.

6. Charge cells at the C/10 rate at 20-23°C.

7. Open the circuit and allow the cells to stand for 168 hours. Measure voltages once each 4 hours for first 12 hours, then once each 24 hours thereafter.

8. After the 168-hour stand, discharge the cells under conditions identical to those used in Step 4. Record ampere-hours discharged.

9. Subtract ampere-hours in Step 8 from ampere-hours in Step 4 and calculate percent capacity retained.

10. Plot voltage data versus the logarithm of the open-circuit stand time in hours. Consider entire curve in evaluation of results. Observe degree of conformance to straight line. Non-linear, downward curvature indicates a significant internal short. Unusually steep negative slope indicates other charge loss mechanisms.

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APPENDIX A

TEST PROCEDURES

FOR

TEST SEQUENCES 1 THROUGH 5

TEST PROCEDURE

FOR

TEST SEQUENCE NO. 1

1. Preparation for Test

1.1 Prepare to record the following:

a) Cell pack current
b) Cell voltages
c) (-) Terminal to case voltages
d) Cell pack temperature
e) Divide the cells in Test Lot No.1 into two groups, designated as groups 1 and 2.

Group	No. of Cells
1	24
2	24

Cells shall be further divided into sub-groups as follows:

Group 1		Cell No.
Subgroup	1A	1 thru 6
	1B	7 thru 12
	1C	13 thru 18
	1D	19 thru 24
Group 2		
Subgroup	2A	25 thru 30
	2B	31 thru 36
	2C	37 thru 42
	2D	43 thru 48

2. Procedure Prior to Acceptance Testing

2.1 All Cells. Remove shorts and record cell voltages at 1 hour intervals for 24 hours.

2.2 Group 1 Cells

a) Following completion of the initial 24 hour open-circuit stand, apply shorts to each cell for 4 $\pm$ 1 hours.

b) Divide cells into sub-groups as shown in Paragraph 1.1.

c) Charge each of these groups at 2.40 $\pm$ 0.10 amperes for the time periods as follows (record cell voltages prior to terminating charge):

Subgroup	Charge Time (minutes)
1A	6 ($\pm$ 10 seconds)
1B	12 ($\pm$ 20 seconds)
1C	12 ($\pm$ 20 seconds)
1D	18 ($\pm$ 30 seconds)

d) Following this charge allow all sub-groups to stand on open circuit for 72 $\pm$ 4 hours. During the first hour of the 72 hour stand attach the following resistors across the designated cells of sub-group 1C: Resistors shall remain attached until the completion of the 72 hour period.

Cells 1 and 2	100 ohms, ½ watt, $\pm$ 5%
Cells 3 and 4	1000 ohms, ½ watt, $\pm$ 5%
Cells 5 and 6	10,000 ohms, ½ watt, $\pm$ 5%

e) During the 72 hour stand period and print-out data at least once each 20 minutes during the first hour, then once per hour for the remainder of the 72 hour period.

f) At the end of the 72 hour stand period, apply shorts to each cell for 4 $\pm$ 1 hours.

g) Remove all shorts and resistors from the cells. Charge all cells at 2.40 $\pm$ 0.10 amperes for 20 $\pm$ 1 hours with the baseplate held at 21 $\pm$ 3°C (70 $\pm$ 5°F). If the voltage of any cell in a series string exceeds 1.50 volts remove that cell from the circuit.

h) Discharge each string at 12.0 $\pm$ 0.50 amperes until the first cell in that string goes below 1.0 volts, then discontinue discharge.

i) Apply ¼ ohm resistors to all cells for 16 $\pm$ 1 hours.

j) Apply shorts to all cells for 4 $\pm$ 1 hours.

k) Charge each sub-group at 2.40 ± 0.10 amperes for the same time periods as in (c).

l) Repeat Paragraphs (d) and (e).

m) At the end of the stand period apply shorts to each cell.

2.3 Group 2 Cells

a) Following completion of the initial 24 hour open-circuit stand, charge all cells in Group 2 at 2.40 ± 0.10 amperes for 20 ± 1 hours, with the baseplate held at 21 ± 3°C (70 ± 5°F). If any cell voltage exceeds 1.50 volts remove that cell from the circuit.

b) Discharge each string at 12.0 ± 0.50 amperes until the first cell in that string goes below 1.0 volts, then discontinue discharge.

c) Apply ½ ohm resistors to all cells in the group for 16 ± 1 hours.

d) Apply shorts to all cells in the group for 4 ± 1 hours.

e) Charge subgroups at 2.40 ± 0.10 amperes for the indicated time periods (record cell voltages prior to terminating charge):

Subgroup	Charge Time (minutes)
2A	6 (± 10 seconds)
2B	12 (± 20 seconds)
2C	12 (± 20 seconds)
2D	18 (± 30 seconds)

f) Following this charge allow cells to stand open-circuit for 72 ± 4 hours. Attach resistors to cells of sub-groups 2C as described for sub-group 1C in Paragraph 2.2 (d).

g) At the end of the stand period apply shorts to the cells for 4 ± 1 hours.

h) Remove all resistors and shorts from the cells and charge cells as specified in Paragraph (e).

i) Repeat Paragraph (f).

j) At the end of the stand period apply shorts to all cells.

3. Procedure After Acceptance Testing

3.1 All Cells. After completing the third of three calibration charge-discharge cycles:

a) Apply ¼ Ω resistors to all cells for 16 ± 1 hours.

b) Remove resistors and allow cells to stand open circuit for 24 ± 1 hours. Record Group 1 cell voltages once per hour during this period. Make no measurements on Group 2 cells.

c) Apply shorts to all cells for 4 ± 1 hours.

3.2 Cell Groups

a) Charge each cell subgroup as follows (record cell voltages prior to terminating charge):

Subgroup	Charge Rate (Amperes)	Charge Time (Minutes)
1A	2.4	6 (± 10 seconds)
1B	2.4	6 (± 10 seconds)
1C	2.4	12 (± 20 seconds)
1D	2.4	12 (± 20 seconds)
2A	12.0	1.2 (± 10 seconds)
2B	12.0	1.2 (± 10 seconds)
2C	12.0	2.4 (± 10 seconds)
2D	12.0	2.4 (± 10 seconds)

b) Following this charge allow all cells to stand for a period of 72 ± 4 hours. Attach the following resistors to cell groups 1A, 1C, 2A, and 2C. Cell groups 1B, 1D, 2B, and 2D shall remain open-circuited.

Cell Groups 1A, 1C, 2A and 2C

Cells 1 and 2	100Ω, ½ watt, ± 5%
Cells 3 and 4	330Ω, ½ watt, ± 5%
Cells 5 and 6	1000Ω, ½ watt, ± 5%

c) During the 72 hour stand period record and printout data once each 20 minutes during the first hour, then once per hour for the remainder of the 72 hour period.

d) At the end of the stand period apply shorts to each cell. This completes the test sequence.

TEST PROCEDURE

FOR

TEST SEQUENCE NO. 2

1. Data Requirements

a) Data to be recorded shall include cell volts, case to negative volts, pack current, and one baseplate temperature for each piece of test equipment used.

b) Record data during charge and discharge, stand with a frequency to providing at least 10 readings during these conditions. During short charges of 6 minutes or less, only one set of readings are required near end of charge. Open circuit voltage readings shall be taken on tape once each hour.

2. Procedure

a) Regulate heat sink temperature at 73 $\pm$ 3°F throughout the test. Remove the shorts and record voltages once per hour for 24 hours.

b) Replace shorts for 4 hours.

c) Remove shorts.

d) Charge six of the 12 cells at 1.2A for 6 minutes. Charge the other six cells at 1.2A for 12 minutes.

e) Put all cells on open circuit. Record voltages once each 20 minutes for the first two hours, then once per hour for a total stand time of 72 hours.

f) At the end of the open circuit stand period, charge the cells at 1.2A for 24 $\pm$ 2 hours. Remove any cell from charge if the voltage reaches 1.50 volts.

g) Discharge the cells at 6.0A. Remove each cell from discharge as the voltage reaches 1.0, +0, -0.1 volt.

h) Apply 0.5 ohm resistors and let stand for 16 hours. Record cell voltages during this time.

i) Remove the resistors and allow the cells to stand on open circuit for 24 hours. Record voltages once per hour during this period.

j) Repeat Paragraphs (b), (c), (d) and (e).

k) Short all cells at end of this time.

TEST PROCEDURE

FOR

TEST SEQUENCE NO. 3

1. Test Articles

The test articles shall consist of 54 Gulton 15 ampere-hour cells disignated as Test Lot No. 3.

2. Preparation for Test

2.1 Cells shall be selected at random and grouped into nine (9) six-cell packs. These shall be designated as Groups 1, 2, and 3, with Subgroups A, B, and C in each group.

2.2 Prepare to record the following on the data acquisition system:

a) Cell pack currents

b) Cell voltages

c) Baseplate temperatures

d) Case to negative voltages

3.0 Test SetUp

3.1 Install cell packs in restraining fixtures. Torque fixture restraining bolts (4) to 20 $\pm$ 2 in-lbs.

3.2 Install cell packs on baseplates and adjust temperature of heat sink to 23 $\pm$ 2°C (73 $\pm$ 5°F). Maintain this baseplate temperature for duration of test.

3.3 Remove shorting wires from all cells.

4.0 Test Procedure

4.1 Impedance Measurements

Select 12 cells at random and measure and record the impedance at 60 Hz between the positive (+) and negative (-) terminals of each.

4.2 Group 1 Tests

a) Charge all cells at a constant current of 0.75 $\pm$ 0.050 amperes for a period of 40 $\pm$ 1 hours. Record charge current and cell voltages a minimum of once every 30 minutes and just prior to terminating charge.

4.2 (Continued)

b) Discharge each cell pack at 7.50 $\pm$ 0.075 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 $^{+0.0}_{-0.2}$ volts.

c) Apply ½ ohm resistors to all cells for 16 $\pm$ 1 hours.

d) Remove resistors and apply shorts to each cell for 4 $\pm$ 1 hours.

e) Remove shorts and allow cells to stand open-circuit for 24 $\pm$ 1 hours. Record cell voltages a minimum of once per hour, except during the first hour when they shall be recorded and printed out every 20 minutes.

f) Re-apply shorts to each cell for a period of 4 $\pm$ 1 hours.

g) Charge each cell pack at 1.5 $\pm$ 0.1 amperes for the following periods of time (record cell voltages prior to terminating charge):

Subgroup	Charge Time (minutes)
1A	3 $\pm$ 0.1
1B	6 $\pm$ 0.2
1C	9 $\pm$ 0.3

h) Following charge allow all subgroups to stand on open circuit for 48 $\pm$ 2 hours. During this period record and printout cell voltages at least once each 20 minutes during the first hour, then once per hour for the remainder of the period.

i) At the end of the 48 hour stand apply shorts to each cell for 20 $\pm$ 4 hours.

j) Remove shorts and charge cells at 1.5 $\pm$ 0.1 amperes for 22 $\pm$ 2 hours.

k) Discharge each cell pack at 7.50 $\pm$ 0.075 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge, Remove individual cells as they readh 1.0 $^{+0.0}_{-0.2}$ volts.

l) Apply ½ ohm resistors to all cells for 16 $\pm$ 1 hours.

m) Remove resistors and apply shorts to each cell for 24 $\pm$ 1 hours.

n) Attach the following resistors to each cell in subgroups indicated. Then remove shorts:

4.2 (Continued)

Subgroup	Resistance (ohms)
1A	No resistor
1B	500, ½ watt, ± 5%
1C	1000, ½ watt, ± 5%

Allow cells to stand in this condition for 48 ± 2 hours. During this period record and printout cell voltages at least once each 20 minutes during the first hour, then once per hour for the remainder of the 48 hour period.

o) At the end of the 48 hour stand, apply shorts to each cell for 20 ± 4 hours.

p) Remove shorts and resistors and charge at 1.5 ± 0.1 amperes for 24 ± 1 hours.

q) Allow cells to stand on open circuit for 1 ± 0.1 hour.

r) Discharge each cell pack at 7.50 ± 0.075 amperes until all cells in each group go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach $1.0\,^{+0.0}_{-0.2}$ volts. Calculate and record A-H capacity to 1.0V for each cell.

s) Apply ½ ohm resistors to all cells for 16 ± 1 hours.

t) Remove resistors and apply shorts to each cell for 4 ± 1 hours.

u) After the shorted stand period is completed and with shorts left installed, attach resistors to each cell of the subgroups indicated:

Subgroup	Resistance (ohms)
1A	No resistor
1B	500, ½ watt, ± 5%
1C	1000, ½ watt, ± 5%

v) Remove shorts but <u>not</u> the resistors and charge cells at 1.5 ± 0.1 amperes for 24 ± 1 hours.

w) Allow cells to stand, with resistors installed, for 7 days (168 ± 8 hours). Record and printout cell voltages once each 20 minutes for the first hour, and then once every four hours for the remainder of the period.

4.2 (Continued)

x) With resistors still installed, discharge each cell pack at 7.50 $\pm$ 0.075 amperes until the last cell in each pack goes below 1.0 volt, then discontinue discharge. Remove cells when $V_C \leq 1.0V$. Calculate and record ampere-hour output to 1.0 volt for each cell.

y) Remove resistors and install ½ ohm resistors across each cell for 16 $\pm$ 1 hours.

z) Remove resistors and apply shorts to each cell.

4.3 Group 2 Tests

a) Charge all cells at a constant current of 0.75 $\pm$ 0.050 amperes for a period of 40 $\pm$ 1 hours. Record charge current and cell voltages a minimum of once every 30 minutes and just prior to terminating charge.

b) Discharge each cell pack at 7.50 $\pm$ 0.075 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 $^{+0.0}_{-0.2}$ volts.

c) Apply ½ ohm resistors to all cells for 16 $\pm$ 1 hours.

d) Remove resistors and apply shorts to each cell for 4 $\pm$ 1 hours.

e) Remove shorts and charge each cell pack at 1.5 $\pm$ 0.1 amperes for the following periods of time (record cell voltages prior to terminating charge):

Subgroup	Charge Time (minutes)
2A	3 $\pm$ 0.1
2B	6 $\pm$ 0.2
2C	9 $\pm$ 0.3

f) Following charge allow all subgroups to stand open circuit for 72 $\pm$ 4 hours. Record and printout cell voltages once each 20 minutes during the first hour, and once per hour for the remainder of the period.

g) At the end of the 72 hour stand apply shorts to each cell for 20 $\pm$ 4 hours.

h) Remove shorts and charge cells at 1.5 $\pm$ 0.1 amperes for 22 $\pm$ 2 hours.

C-3

4.3 (Continued)

i) Discharge each cell pack at 7.50 ± 0.075 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 $^{+0.0}_{-0.2}$ volts.

j) Apply ½ ohm resistors to all cells for 16 ± 1 hours.

k) Remove resistors and apply shorts to each cell for 4 hours.

l) Attach the following resistors to each subgroup and then remove shorts:

Subgroup	Resistance (ohms)
2A	2000, ½ watt, ± 5%
2B	No resistor
2C	500, ½ watt, ± 5%

m) Charge cells at 1.5 ± 0.1 amperes (with resistors installed) for 6 minutes.

n) Allow cells to stand in this condition for 48 to 72 hours. During this period record cell voltages at least once each hour.

o) At the end of the 48 to 72 hour stand apply shorts to each cell for 20 ± 4 hours.

p) Remove shorts and resistors and charge at 1.5 ± 0.1 amperes for 24 ± 1 hours.

q) Allow cells to stand on open circuit for 1 ± 0.1 hours. Record and printout cell voltages once each 20 minutes.

r) Discharge each cell apck at 7.50 ± 0.075 amperes until all cells in each group go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 $^{+0.0}_{-0.2}$ volts. Calculate and record A-H capacity to 1.0 volts for each cell.

s) Apply ½ ohm resistors to all cells for 16 ± 1 hours.

t) Remove resistors and apply shorts to each cell for 4 hours.

u) After the shorted stand period is completed and with shorts left installed, attach the following resistors:

4.3 (Continued)

Subgroup	Resistance (ohms)
2A	2000, ½ watt, ± 5%
2B	No resistor
2C	500, ½ watt, ± 5%

v) Remove shorts but not the resistors and charge cells at 1.5 ± 0.1 amperes for 24 ± 1 hours.

w) Allow cells to stand, with resistors installed, for 7 days (168 ± 8 hours). Record and printout cell voltages every 20 minutes during the first hour, and then once every four hours for the remainder of the period.

x) With resistors still installed, discharge each group at 7.50 ± 0.075 amperes until the last cell goes below 1.0 volt, then discontinue discharge. Record ampere hour output to 1.0 volt for each cell. Remove individual cells as they reach 1.0 volts.

y) Remove resistors and install ½ ohm resistors across each cell for 16 ± 1 hours.

z) Remove resistors and apply shorts to each cell.

4.4 Group 3 Tests

a) Charge all cells at a constant current of 0.75 ± 0.050 amperes for a period of 40 ± 1 hours. Record charge current and cell voltages a minimum of once every 30 minutes and just prior to terminating charge.

b) Discharge each cell pack at 7.50 ± 0.075 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 volts.

c) Apply ½ ohm resistors to all cells for 16 ± 1 hours.

d) Remove resistors and apply shorts to each cell for 22 ± 2 hours.

e) Remove shorts and charge each cell pack at 1.5 ± 0.1 amperes for the following periods of time (record voltages prior to terminating charge):

Subgroup	Charge Time (minutes)
3A	3 ± 0.1
3B	6 ± 0.2
3C	9 ± 0.3

4.4 (Continued)

f) Following the charge allow all subgroups to stand on open circuit for 72 $\pm$ 4 hours. Record and printout cell voltages every 20 minutes during the first hour, and then once per hour for the remainder of the period.

g) At the end of the 72 hour stand apply shorts to each cell for 20 $\pm$ 4 hours.

h) Remove shorts and charge cells at 1.5 $\pm$ 0.1 amperes for 22 $\pm$ 2 hours.

i) Discharge each cell pack at 7.50 $\pm$ 0.075 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 volts.

j) Apply ½ ohm resistors to all cells for 16 $\pm$ 1 hours.

k) Remove resistors and apply shorts to each cell for 4 hours.

l) Attach the following resistors to each cell in each subgroup and then remove shorts:

Subgroup	Resistance (ohms)
3A	1000, ½ watt, $\pm$ 5%
3B	2000, ½ watt, $\pm$ 5%
3C	No resistor

m) Charge cells at 1.5 $\pm$ 0.1 amperes (with resistors installed for 6 minutes.

n) Allow cells to stand in this condition for 48 hours. During this period record and printout cell voltages once each hour, except during the first hour when it shall be every 20 minutes.

o) At the end of the 48 hour stand apply shorts to each cell for 24 + 4 hours.

p) Remove shorts and resistors and charge at 1.5 $\pm$ 0.1 amperes for 24 $\pm$ 1 hours.

q) Allow cells to stand on open circuit for 1 $\pm$ 0.1 hours. Record and printout cell voltages once each 20 minutes.

4.4 (Continued)

r) Discharge each cell pack at 7.50 ± 0.075 amperes until all cells in each group go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 volts. Measure and record A-H capacity to 1.0 volt for each cell.

s) Apply ½ ohm resistors to all cells for 16 ± 1 hours.

t) Remove resistors and apply shorts to each cell for 4 hours.

u) After the shorted stand period is completed and with shorts left installed, attach the following resistors:

Subgroup	Resistance (ohms)
3A	1000, ½ watt, ± 5%
3B	2000, ½ watt, ± 5%
3C	No resistor

v) Remove shorts but not the resistors and charge cells at 1.5 ± 0.1 amperes for 24 ± 1 hours.

w) Allow cells to stand, with resistors installed, for 7 days (168 ± 8 hours). Record and printout cell voltages once every 20 minutes for the first hour, and then once every 4 hours for the remainder of the period.

x) With resistors still installed, discharge each cell pack at 7.50 ± 0.075 amperes until the last cell in each pack goes below 1.0 volt, then discontinue discharge. Measure and record ampere-hour output to 1.0 volt for each cell. Remove individual cells as they reach 1.0 volts.

y) Remove resistors and install ½ ohm resistors across each cell for 16 ± 1 hours.

z) Remove resistors and apply shorts to each cell. This completes the test sequence.

TEST PROCEDURE
FOR
TEST SEQUENCE NO. 4

1. Test Articles

The test articles shall consist of twelve General Electric 12 AH cells designated as Test Lot No. 4.

2. Preparation for Test

2.1 Cells shall be selected at random and grouped into two six-cell packs designated as Group 1 and Group 2.

2.2 Each group shall be secured separately in a restraining fixture.

3. Test SetUp

3.1 Prepare to record the following on the data acquisition system:

a) Cell pack currents
b) Cell voltages
c) Baseplate temperature
d) Case to negative terminal voltages

During open circuit stand periods record data once every 20 minutes during the first hour, once per hour during the second, third, and fourth hours, and then once every four hours for the remainder of the period.

3.2 Install cell packs in restraining fixtures and torque the four restraining bolts to 20 $\pm$ 2 in-lbs.

3.3 Install cell packs on baseplate and adjust temperature of heat sink to 21 $\pm$ 3°C (70 $\pm$ 5°F). Maintain this baseplate temperature for duration of test.

4. Test Procedure

4.1 Remove shorting wires from individual cells.

4.2 Immediately after removing shorting wires record and printout open circuit cell voltages.

4.3 Disable data acquisition system and measure the impedance at 60 Hz between the positive (+) and negative (-) terminals of each cell. Record results and calculate and record cell impedances.

4.4 Re-enable data acquisition system and record open circuit cell voltages.

4.5 Allow cells to remain open circuit for a period of 24 ± 1 hours following removal of shorting wires. Record data in accordance with Paragraph 3.1 during this period.

4.6 Charge all cells at a constant current of 1.2 ± 0.1 amperes for the following periods of time. Measure and record charge current and cell voltages prior to terminating charge:

Group 1:	3 ± 0.1 minutes
Group 2:	6 ± 0.2 minutes

4.7 Following charge allow all cells to stand on open circuit for 72 ± 4 hours. During this period record and printout data per Paragraph 3.1.

4.8 At the end of the 72 hour stand period, apply shorts to individual cells for 4 to 16 hours.

4.9 Remove shorts and charge cells at 1.2 ± 0.1 amperes for 24 ± 2 hours. Record data a minimum of once per hour during this period.

4.10 Discharge each cell pack at 6.0 ± 0.06 amperes until all cells in each pack go below 1.0 volt, then discontinue discharge. Remove individual cells as they reach 1.0 volt. Record data a minimum of once every 30 minutes during this period.

4.11 Attach ½ ohm resistors to each cell for 16 ± 2 hours. All cell voltages must be ≤ 0.1 volt at the conclusion of this period. Record data a minimum of once per hour during this period.

4.12 Remove resistors and short cells for a period of 4 ± 1 hours.

4.13 Remove shorts and allow cells to stand open circuit for a period of 24 ± 2 hours. During this period record and printout data per Paragraph 3.1.

4.14 At the end of the 24 hour stand period apply shorts to individual cells for 4 ± 1 hours.

4.15 Remove shorts and charge cells at a constant current of 1.2 ± 0.1 amperes for the following periods of time. Measure and record charge current and cell voltages prior to terminating charge:

Group 1: 3 ± 0.1 minutes

Group 2: 6 ± 0.2 minutes

4.16 Following charge all cells to stand on open circuit for 72 ± 4 hours. During this period record and printout data per Paragraph 3.1.

4.17 At the end of the 72 hour stand period repeat the impedance measurements of Paragraph 4.3.

4.18 Apply shorts. This concludes this test sequence.

TEST PROCEDURE

FOR

TEST SEQUENCE NO. 5

1. PURPOSE OF TEST

To provide data showing the short test response of 50 Ah cells to compare with data from smaller cells, to provide direct comparative data between cells with polypropylene separators and those with nylon separators; to provide additional data comparing the 24 hour test with the 7 day charged stand test; and to obtain data relative to the above with cells containing built in reference electrodes in order to identify which electrode is determining cell voltage under open-circuit conditions.

2. TEST ARTICLES

The test articles shall consist of forty-eight 50 Ah cells designated as Test Lot No. 5.

3. PREPARATION FOR TESTING

3.1 Set-Up

Arrange cells in eight packs of six-cells each. Each pack shall contain 3 cells each with nylon and polypropylene separators. Bond six-packs to baseplates. Make electrical connections, including power supply, series current path through each six pack. Data as follows:

a) Cell Terminal Voltage
b) Anx. No. 2 (O_2 electrode) to Negative Terminal Voltage
c) Reference Electrode to Negative Terminal Voltage
d) Pack Current (one per pack)
e) Baseplate Temperature (2 per pack)

3.2 Data Acquisition

Scan cells for voltage at standard intervals during charge and discharge. During open circuit stand, scan each 20 minutes during the first hour, each hour for the next three hours, then once each four hours thereafter.

3.3 Temperature Control

Operate with fixed circulating fluid temperatures. Start at 70°F. Raise or lower fluid temperature in 5°F increments to maintain baseplate within prescribed limits (70 ± 5°F).

4. TEST PROCEDURE

4.1 Procedure for Packs 1 through 4

a) Charge at 2.5 A for 44 ± 4 hours. Discharge at 25 A to 1.1 volt each cell.

b) Attach 0.1 ohm 25 watt resistors on packs 1-4 until voltages are less than 0.3 volts. Then attach shorts. Total time on resistor and shorts shall be 16 ± 1 hours.

c) Remove shorts and resistors, and allow cells to stand on open circuit for 24 hours.

d) Apply shorts for 60 hours.

e) Remove shorts and charge at 5.0A for 24 ± 1 hours. Place cells on open circuit for 1 hour. Discharge at 25A to 1.1 volt each cell.

f) Attach 0.1 ohm resistors. Allow to stand 16 ± 1 hours.During the time on 0.1 ohm resistors, add the following resistors in parallel to the designated cells (Resistance tolerance ± 10 percent).

Pack 1	None
2	1000 ohm
3	500 ohm
4	100 ohm

g) At the end of the 16 hour period, remove the 0.1 ohm resistors (leaving the higher value resistor in place). Allow to stand for 24 hours.

h) Install shorts for 4 hours.

i) Remove shorts. Charge at 5.0 A for 6.0 minutes. Place cells on open circuit and allow to stand for 100 hours.

j) Install 0.25 ohm resistors on all cells. Allow cells to stand in this manner for 4 days.

k) Remove all resistors from all cells. Charge all cells at 5.0 A for 24 ± 1 hours. At the end of the charge, allow all cells to stand open circuit for 1 hour then discharge all cells at 25.0 A to 1.1 volts.

l) Apply 0.25 ohm resistors. While low-value resistors are on, add the following resistors in parallel:

Pack 1	100 ohms
2	50 ohms
3	20 ohms
4	250 ohms

Allow the cells to stand with these resistors in place for 24 hours.

m) At the end of 24 hours remove the 0.25 ohm resistors, leaving the other resistors in place. Charge at 5.0 A for 6.0 minutes.

n) Put cells on open circuit and allow to stand 48 hours.

o) At the end of the open circuit period, attach 0.25 ohm resistors. Allow to stand for 72 hours.

p) Remove all resistors. Allow cells to stand on open circuit for 24 hours.

q) Charge all cells at 5.0 A for 24 ± 1 hours. After the charge let cells stand on open circuit for 1 hour. Discharge at 25.0 A until the last cell goes below 1.1 volt.

r) Attach 0.25 ohm resistors. Then change the parallel high resistances to be as follows:

Pack 1	None (no change)
2	250 ohms
3	None
4	250 ohms

Allow the cells to stand with the 0.25 ohm resistor in place for 40 hours.

s) Remove the 0.25 ohm resistors from all cells. Charge Packs 1 and 2 at 5.0 A for 6 minutes. Then put these two packs on open circuit for 72 hours. Allow packs 3 and 4 to stand on open circuit for 72 hours.

t) After the open circuit stand attach 0.25 ohm resistors to all cells. This completes the test sequence for packs 1-4.

4.2 Procedure for Packs 5 through 8

(a) through (e): Same as 4.1 (a) through (e).

(f) Apply 0.1 ohm resitors. Allow cells to stand 16 hours.

(g) During the time the low resistors are on, add the following resistors in parallel to the designated cells:

Pack 5	None
6	250 ohms
7	150 ohms
8	50 ohms

(h) At the end of the 16 hour period, remove the 0.1 ohm resistors. All cells to stand for 24 hours.

(i) Install 0.1 ohm resistors for 6 hours.

(j) Remove the 0.1 ohm resistors. Allow cells to stand for 72 hours.

(k) Attach 0.1 ohm resistors and allow to stand 96 hours.

(l) Remove resistors. Charge cells at 5.0 A for 24 $\pm$ hours. Allow cells to stand open circuit for 1 hour. Then discharge at 25 A to 1.1 volts each cell.

(m) Apply 0.1 ohm resistors. While these resistors are in place, add resistors as indicated:

Pack 5	100 ohms
6	50 ohms
7	20 ohms
8	10 ohms (Later changed to 250 ohms)

Allow cells to stand with these resistors in place for 24 hours.

(n) Remove the 0.1 ohm resistors and allow the cells to stand for 48 hours.

(o) Replace the 0.1 ohm resistors. Allow to stand for 72 hours.

(p) Remove all resistors. Then attach resistors as indicated:

Pack	5	None
	6	500 ohms
	7	250 ohms
	8	100 ohms

(q) Charge cells at 5.0A for $\pm$ 24 hours. At the end of charge, allow cells to stand 1 hour on open circuit. Discharge at 25 A to 1.1 volts each cell. Compute and record capacities.

(r) Apply 0.1 ohm resistors. Allow to stand 40 hours.

(s) Remove 0.1 ohm resistors. Charge at 5.0 A for 24 $\pm$ hours.

(t) Put cells on open circuit and allow to stand for 168 $\pm$ 4 hours (7 days).

(u) After 168 hours on open circuit, discharge cells at 25A to 1.1 volts. Compute and record capacities.

(v) Attach 0.1 ohm resistors to all cells. This completes the test sequence for Packs 5-8.